ADVANCED TECHNOLOGY LABORATORIES

6 9P

N63 21312
Code 1

NASA CR 51332

PROGRESS REPORT

DEVELOPMENT OF LOW TEMPERATURE DIELECTRIC COATINGS FOR ELECTRICAL CONDUCTORS

ANNUAL SUMMARY AND 8TH QUARTERLY REPORT

BY

K.N. MATHES

JULY 15, 1963

OTS PRICE

XEROX $ 6.60 ph

MICROFILM $ 2.27 mf.

Title Suppl (Out Copy)

ANNUAL SUMMARY AND 8th QUARTERLY REPORT

JULY 15, 1963

Title DEVELOPMENT OF LOW TEMPERATURE DIELECTRIC COATINGS FOR ELECTRICAL CONDUCTORS

(NASA Contract ~~No.~~ NAS8-2442)
Control Number: TP85-498(CPB-02-1096-61)
(W/O #8500-0-0224-000-8500-11-431)

(NASA CR-51332) OTS: $6.60ph, $2.27mf

General Electric Company
Missile and Space Vehicle Department
Philadelphia, Pa.

Report Prepared by:

K. N. Mathes July 15, 1963 69p
General Engineering Laboratory
C.A. General Electric Company
Schenectady 5, N.Y.

3540002

Report Prepared for:

George C. Marshall Space Flight Center
Huntsville, Alabama

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF PHOTOGRAPHS

Annual Summary and 8th Quarterly Report

July 15, 1963

DEVELOPMENT OF LOW TEMPERATURE DIELECTRIC COATINGS FOR ELECTRICAL CONDUCTORS

INTRODUCTION

This report includes a summary of the technical accomplishment under the subject contract during the 8th quarter (April, May and June, 1963) as well as a review of earlier work which together constitute a summary report for the work during the preceding year. Reference is made here to quarterly reports dated October 15, 1962, January 15, 1963 and April 15, 1963, since they contain detailed information which may be only in part summarized in this report. Attention is drawn, also, to the 4th Quarterly Report dated July 16,1962. This report provides a summary of the first year's effort on which the subject work is based.

The subject report includes, as an appendix, a summary of visits made to 16 laboratories in six European Countries in reference to cryogenic properties of materials. A full report of these visits will be issued separately as soon as possible.

The primary activity of the second year's program has involved the evaluation of insulated wire after thermal aging in air and vacuum and after moisture exposure. Such evaluation is very important since these ambients may adversely effect insulation performance at both normal, elevated and cryogenic temperatures. The aromatic, polyimide (DuPont ML) and inorganic bonded, felted asbestos insulated wires (developed in the previous program primarily for cryogenic applications) have been evaluated along with more conventional polyvinylformal (Formvar), polyvinyl chloride (PVC) and polytetrafluoroethylene (Teflon TFE) insulated wires for comparison.

Investigation has been directed, also, to the development of "ribbon" cable suitable for application at cyrogenic temperatures. It is proposed to continue this work in the program for the coming year and to extend it to bundled and cabled constructions also.

During the past year electrical measurements of the cryogenic liquids themselves have been continued because the electrical properties of the liquids have an important relationship to the performance of insulated wire and other components immersed in them.

SUMMARY AND CONCULSIONS

Wire for Cryogenic Applications

The program for the past year confirms the belief that the polyimide polymers (ML and enamel and H film) possess remarkably superior properties at cryogenic temperatures. However, no one material is likely to meet every application problem. For example, Teflon has outstanding advantages where low dielectric losses are needed. Inorganic spacers, such as aluminum phosphate impregnated, felted asbestos, possess advantages under intense radiation or where high temperature, cut-through problems are involved although ML also possesses tremendous resistance to thermal cut-through and excellent resistance to radiation for an organic material.

It is hoped that the data in this report will be useful in selecting wire insulations to meet specific combinations of needs. The advantages and disadvantages of several types of wire insulation are summarized below, keeping in mind not only the cryogenic requirements but other parameters of the application problem also.

ML

Advantages:
- Greatest flexibility at cryogenic temperatures.
- Excellent thermal stability
- No measurable thermal cut-through
- Mechanical toughness

Disadvantages:
- Available only as a relatively thin, film coating
- Some sensitivity to moisture

HF

Advantages:
- Mechanical toughness

Disadvantages:
- Available only as a relatively thin, film coating
- Poor flexibility at cryogenic temperatures
- Sensitivity to moisture
- Thermal stability inferior to ML or Teflon

Extruded Teflon

Advantages:
- Low electrical losses
- Best flexibility at cryogenic temperatures for an extruded insulation
- Resistance to moisture
- Thermal stability
- Resistance to abrasion

Disadvantages:
- Poor flexibility at cryogenic temperatures as compared to ML
- Relatively poor thermal cut-through characteristics
- Poor resistance to radiation

Extruded PVC

Advantages: Resistance to moisture

Disadvantages: Shatter brittle at cryogenic temperatures
Extremely poor thermal cut-through
Poor stability, particularly in vacuum
Relatively high electrical losses

Aluminum Phosphate Impregnated, Felted Asbestos

Advantages: Excellent thermal stability
Completely inorganic composition with associated heat and moisture resistance
Fairly good flexibility at cryogenic temperatures

Disadvantages: Extremely poor resistance to moisture
Low breakdown voltage
High electrical losses (except at cryogenic temperatures)
Poor abrasion resistance

Much more detailed information is, of course, available in the body of this report. It should be remembered, also, that for certain applications insulating materials may be combined to give optimized characteristics. For example, felted asbestos has been applied over an ML film. The combination combines the excellent voltage breakdown of the ML film with the advantages of the inorganic spacing provided by the asbestos. However, the electrical losses of such a combination under normal conditions will be very high, particularly after exposure to moisture. On the other hand, at cryogenic temperatures the losses will be quite low but not nearly as low as for Teflon.

Ribbon Cable for Cryogenic Applications

Ribbon cable, by virtue of its thin construction, has an inherent advantage for use at cryogenic temperatures. However, much remains to be done in optimizing its construction. The use of H film, perhaps bonded with FEP Teflon seems to provide the most fruitful approach.

Vacuum as an Application Parameter

The vacuum of outer space has been thought by some to be a very difficult application parameter. In listening to such discussions, one might come to the belief that high vacuum "sucks the life blood" out of insulating materials. On the other hand, others have indicated that high vacuum must markedly improve the dielectric performance of electrical insulation because high vacuum itself is an excellent insulator.

The results of the subject work lead to the conclusion that neither of the extreme views above is correct. While moderate vacuum (not necessarily high vacuum) at elevated temperatures may degrade PVC by increasing the loss of volatile plasticizers, the same vacuum will increase the thermal life of materials like Formex in which thermal degradation is accelerated by oxidation. Vacuum greatly increases the voltage breakdown of fibrous insulation but somewhat decreases the voltage breakdown of extruded and film-coated insulations. The reasons for the increased breakdown are easy to understand. The decreased breakdown voltage is not yet understood and much more study is needed. Such results provide a note of caution for those who believe that vacuum will in itself solve difficult insulation problems.

Cryogenics and the Theory of Dielectrics

Perhaps the greatest long-run value of the subject program may lie in the clues provided for more basic understanding of insulating materials (dielectrics) even though such clues are not a part of the basic purpose of this program. Both mechanical and electrical measurements made at cryogenic temperatures appear to provide very sensitive means for determining and understanding the changes brought about in insulating materials as a result of the influence of heat and moisture, for example.

The study of dissipation factor and capacitance of cryogenic liquids has provided basic scientific information not before available. The need for additional study has become obvious. Cryogenic liquids, particularly helium, in the normal and super fluid states, offer opportunities for fascinating theoretical studies. Out of such studies results of practical importance will also undoubtedly develop.

Cryogenics in Europe

It is apparent from the author's visits that no program like the subject program exists anywhere in Europe, except possibly in the Soviet Union. At the same time, great interest in the subject work was apparent everywhere. It must be remembered that outside of the Soviet Union, interest in space problems and cryogenic liquids as fuels exists only in France and in Great Britain. The latter country is closely tied to the work in the United States and has not yet, at least, started engineering evaluations.

However, theoretical dielectric studies of high competence are being directed particularly to cryogenic liquids and in some cases, to nonmetallic materials at cryogenic temperatures. Without question, these theoretical studies have a great importance in reference to the program of this report. The studies of voltage breakdown in cryogenic liquids by Lewis in London and Blaisse in Delft, as well as the ion migration studies in liquid helium by Carerri in Rome can be quoted in illustration. The dielectric loss studies in glass and quartz by Volger at Eindhoven and the stress-strain curves for plastics in liquid nitrogen by Dubois in Paris are important, also. Correlary cryogenic information has been obtained in Europe, also. The determination of impurities and the means of purification for cryogenic liquids can be mentioned in example.

OBSERVATIONS AND SUMMARY OF TEST RESULTS

The round wire insulations evaluated in this report are described in Table I. The ribbon wire constructions are described in Table II. The development of ribbon wire for cryogenic applications so far is limited and evaluation has been restricted to repeated mandrel flexibility tests in liquid helium.

The extensive evaluation of round wire insulation has been limited to the following wire insulations:

HML -- Developed for cryogenic applications
Asbestos (phosphate) - " " " "
HF -- For comparison
Teflon - For comparison
PVC -- For comparison

Less extensive investigations have been carried out with the other wires listed in Table I. The full range of the investigations as accomplished is outlined in Table III.

The range of tests actually accomplished is considerably more extensive than required by contract although in a few instances, some conditions as originally requested have not been met. In such cases, the progress of the work indicated either that the specific test results were not needed or that the test techniques were not adequate under the particular test conditions. The principal differences between the evaluation work accomplished and that specified are listed below:

1. Samples aged 20 days in vacuum at 120 C and 120 days in air at 250 C have been added.

2. Breakdown voltage after aging has been measured not only at 23 C as specified but also after immersion in liquid helium (-269 C).

3. Volume resistance, rather than insulation resistance has been measured to permit more precise understanding of the test results. Both volume and surface resistance have been measured at 23 C and in liquid nitrogen (-196 C) but only after moisture exposure since, except for asbestos, the values for the dry samples were too high for accurate measurement. Measurements at 23 C were made at 95% RH to prevent drying.

4. Dielectric constant cannot be measured with the cabled test sample which was the only adequate sample so far with which tests could be made in liquid helium. Capacitance values were measured instead. Capacitance and dissipation factor have not been measured at 120 and 250 C because thermoplastic flow of the cabled sample at these temperatures would make results meaningless. By oversight, the author did not arrange for such tests using different techniques.

Table II

Description of Ribbon Wire Constructions

Sample No.	Material
A	Methode Plyoduct (PD-812-P4) (31/32" Wide x 0.012" Thick, 12 Copper Conductors) (Mylar)
B	Polystrip (H-100-C-25) (2-5/8" Wide x .0086" Thick, 25 Conductors) (Resin Bonded H Film)
C	Polystrip (P-100-C-12) 1-5/16" Wide x .0085" Thick, 12 Conductors) (Resin Bonded Mylar)
D	Polystrip (TX-156-C-20) (3¼ Wide x .012" Thick, 20 Conductors) (FEP Teflon)
E	IRC-HX-100-C-12 (1-5/16" Wide x .012" Thick, 12 Conductors) (.002 H Film/.002 FEP Teflon - .005 FEP Teflon - heat bonded)

Table III

Scope of Tests

	Test Conditions											Aging Before Test								
	-269C		-253C	-196C	-60C	23C		120C		250C										
	In Liq. Helium	In Vacuum	In Liq. Hydrogen	In Liq. Hydrogen	In Air	In Air	In Vacuum	In Air	In Vacuum	In Air	In Vacuum	As Received	15 Days at 80^oC -95% RH	20 Days at 120C in vac.	60 Days at 120 C in vac.	120 Days at 120 C in vac.	60 Days at 120 C in air	120 Days at 120 C in air	60 Days at 250^oC in air	120 Days at 250^oC in air
Breakdown Voltage	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	x								
	✓					✓							x	x	x	x	x	x	x	x
Capacitance and Dissipation Factor	✓			✓		✓						x								
	✓			✓		✓								x	x	x	x	x	x	x
				✓		✓							x							
Volume Resistance				✓		✓							x							
Surface Resistance				✓		✓							x							
Repeated Mandrel Flexibility	✓			✓	✓	✓						x								
	✓					✓								x	x	x	x	x	x	x
Compression and Shatter Resistance				✓		✓		✓		✓		x								
Abrasion Resistance						✓						x								

NOTE: Vacuum was held at 10^{-5} Torr or less.

5. Repeated, reverse, mandrel flexibility tests have been made as specified and also substituted for the single bend tests which were found to give less reliable results at cryogenic temperatures. Flexibility tests were not made in liquid hydrogen (-253C) since no essential difference was noted between tests made in liquid nitrogen (-196C) and in liquid helium (-269C). Tests, however, have been made at -60C.

Voltage Breakdown as a Function of Ambient Test Condition

All voltage breakdown measurements have been made using the short time test technique of ASTM D149 with NEMA twisted pair test specimens. A comparative summary of test results at different temperatures in various mediums (i.e., air, vacuum and cryogenic liquid) is given for film-coated wires in Table IV A and for the other wires in Table IV B.

Since it is cumbersome to compare results in table form, a number of figures have been prepared to illustrate the significant observations which may be drawn from the results. The ratio of the average breakdown voltage at a specified test condition to the value for the wire as received in air at 23C - 50% RH is shown in Figs. 1, 2 and 3.

From Fig. 1 it is apparent that the breakdown voltage of extruded Teflon is not significantly influenced by test temperatures from -269C to 250C nor by vacuum. In contrast, the breakdown voltage of PVC is markedly decreased at 120C in both air and vacuum.

The decrease in breakdown voltage of PVC in vacuum at room temperature is particularly interesting. It might be expected that in such "hard" vacuums, gas ionization (corona) around the wires would be prevented and thereby breakdown voltage would be increased. Quite the contrary result has been obtained and similar results will be seen also in Fig. 2. Dr. L.J. Frisco in work at Johns Hopkins has noted similar reductions in voltage breakdown strength of plastics in hard vacuum. No rigorous explanation has so far been advanced. Electron emission from metal electrodes has been proposed as the mechanism of breakdown in hard vacuum but this mechanism can hardly explain the decreased values obtained. It may be conjectured that the air pressure at the surface of the test specimen is higher than in the surrounding space -- perhaps due to outgassing. Such an hypothesis is supported by the relatively small change of breakdown voltage, if any, which occurs in vacuum at liquid helium temperatures where outgassing is not likely to occur. Moreover, the outgassing of Teflon is known to be minimal so that maintenance of high breakdown voltage in vacuum with Teflon can be similarly explained.

Fig. 2 compares the breakdown voltage of film-coated wires including felted asbestos over HML enamel. Reference to Table IV A will show that the breakdown voltage of the HML asbestos combination is relatively low in air at room temperature. It may be postulated that the asbestos covering has a low resistance which distributes the applied voltage to the relatively weaker points of the ML coating or acts to increase the

Table IV-A

Breakdown Voltage of Film Coated Wire (Kilovolts)
As a Function of
Ambient Test Condition

Wire	Avg. Wall Thick.-In.		-269C Liq. H_e	-269C Vac.	-253C Liq. H_2	-196C Liq. N_2	-60C Air	23C Air	23C Vac.*	120C Air	120C Vac.*	250C Air	250C Vac.*
			7.9	7.4	7.4	8.3	6.4	6.8	5.4	6.1	6.1	2.3	4.9
HF	.0013		8.0	7.7	8.5	9.2	8.8	8.8	5.8	7.0	6.2	3.2	4.9
			8.1	8.4	8.7	9.2	9.3	—	6.5	8.0	6.4	4.1	5.0
		Avg.	8.0	7.8	8.2	8.9	8.2	7.8	5.9	7.0	6.3	3.3	4.9
			5.8 5.3	5.5 5.2	5.7	5.7 5.6		6.1 5.6					
HML(GE)	.0010		6.3 5.5	5.7 5.3	6.7	6.1 6.2		6.2 5.8					
			6.3 5.9	6.1 5.7	6.9	6.7 6.9		6.7 5.9					
		Avg.	6.1 5.6	5.8 5.4	6.4	6.2 6.2		6.3 5.8					
			7.2 7.9	7.4 7.4	8.6 7.4	8.8 8.3	10.9	9.8 11.8	7.5	12.1	6.3	7.4	5.4
HML(GE)	.0014		7.7 8.0	7.5 7.7	9.0 8.5	9.2 9.2	11.5	10.3 13.8	7.7	12.3	7.8	7.7	5.8
			8.6 8.1	7.5 8.4	9.3 8.7	9.2 9.2	13.0	10.7 14.6	8.2	14.1	8.1	7.7	5.9
		Avg.	7.8 8.0	7.5 7.8	9.0 8.2	9.1 8.9	11.8	10.3 13.3	7.8	12.8	7.4	7.6	6.0
			5.0				5.8	10.7	5.3	5.3	5.9	5.5	2.6
HML(PD)	.00105		7.0				6.1	11.7	6.0	5.4	5.9	6.5	3.3
			9.6				8.3	—	6.5	7.0	6.3	7.0	3.5
		Avg.	7.2				7.1	11.2	5.9	5.9	6.0	6.3	3.1
Triple			10.8				10.0	11.2	7.4	10.7	7.5	5.6	4.0
ML(PD)	.0016		11.2				11.5	14.6	7.5	11.5	7.7	8.0	4.3
			11.5				—	15.1	8.1	13.8	8.4	11.5	4.6
		Avg.	11.2				10.8	13.6	7.7	12.1	7.9	8.3	4.3
			5.8	5.4	7.1	7.6		4.3					
HML Asbestos	.0069		6.0	5.6	7.7	7.7		4.8					
			6.1	5.7	7.8	8.3		5.9					
		Avg.	6.0	5.6	7.3	7.9		5.0					
			6.7	7.8	8.5	8.8		8.8					
HYT	.0014		8.9	8.2	9.6	9.9		9.1					
			9.1	8.5	10.2	10.6		9.8					
		Avg.	8.2	8.3	9.4	9.8		9.2					

* A vacuum of about 10^{-5} Torr was obtained

Table IV-A (Continued)

Breakdown Voltage of Film Coated Wire (Kilovolts)
As a Function of
Ambient Test Condition

Wire	Avg. Wall Thick.-In.		-269C		-253C	-196C	-60C	23C		120C		250C	
			Liq. H_e	Vac.	Liq. H_2	Liq. N_2	Air	Air	Vac.*	Air	Vac.*	Air	Vac.*
Teflon				6.1	7.8	7.2		5.9					
Suspensoid	.0015		6.2	7.7	8.1	7.7		5.9					
			6.4	8.4	8.8	8.2		6.4					
		Avg.	6.3	7.4	8.2	7.7		6.1					
Nylon			3.0	4.6		4.5		4.0					
Enamel	.0008		3.1	4.7		5.2		4.0					
			3.7	4.9		5.4		4.3					
		Avg.	3.3	4.7		5.0		4.1					
Fused			2.6	3.8		3.8		3.4					
Nylon	.0007		3.1	4.4	4.7	3.8		3.4					
			4.5	4.7	4.7	4.7		3.4					
		Avg.	3.4	4.3	4.7	4.1		3.4					

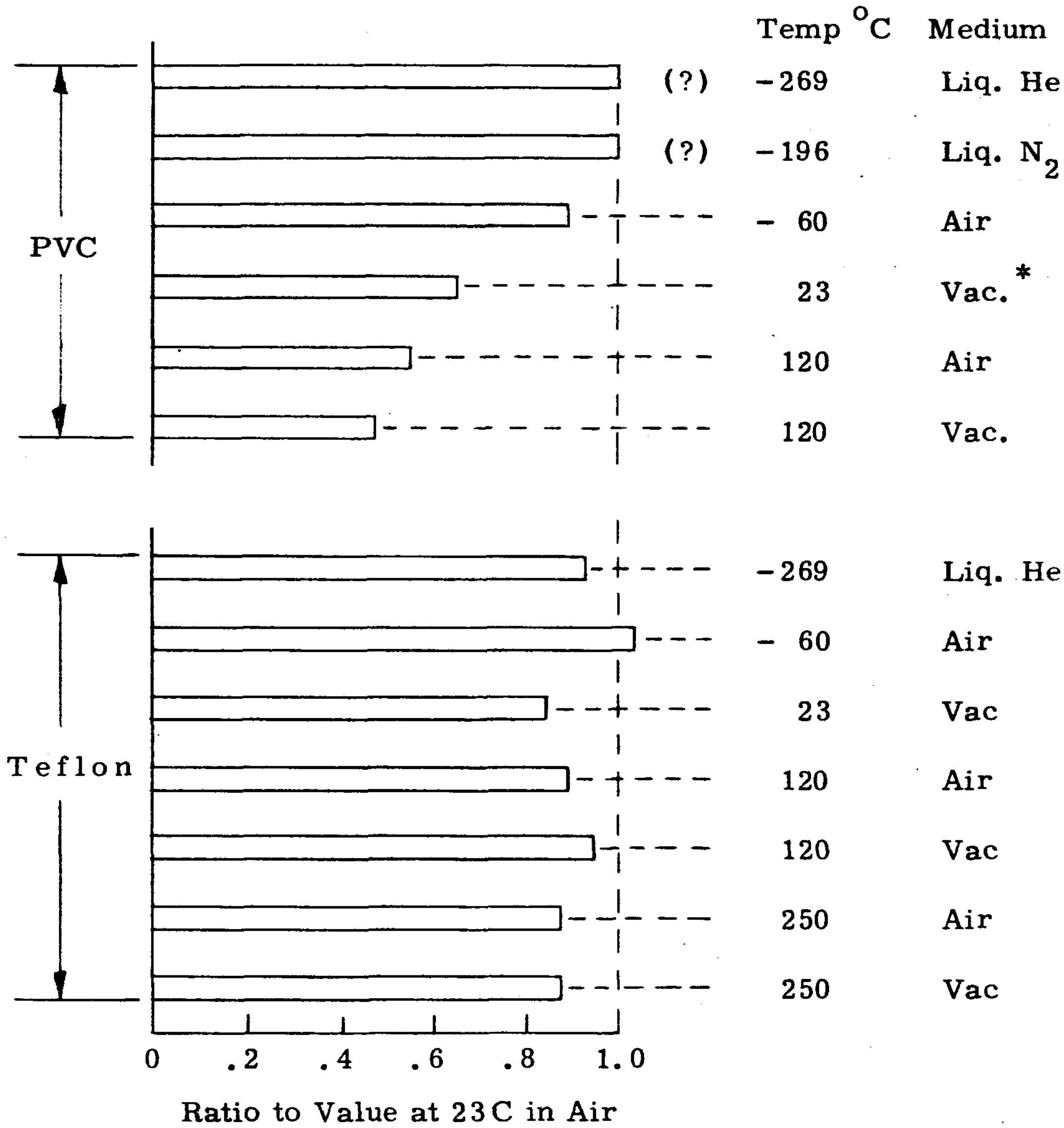
* A vacuum of about 10^{-5} Torr was obtained

Table IV-B

Breakdown Voltage of Extruded and Fibrous Cooled Wire (Kilovolts)
As a Function of
Ambient Test Condition

Wire	Avg. Wall Thick.-In.		-269C Liq. He	-269C Vac.	-253C Liq. H_2	-196C Liq. N_2	-60C Air	23C Air	23C Vac.*	120C Air	120C Vac.*	250C Air	250C Vac.*
			> 20.6			> 20.6	16.8	> 20.6	13.5	11.0	10.0		
PVC	.0071		> 20.6			> 20.6	18.0	> 20.6	14.0	12.0	10.3	Not	
			> 20.6			> 20.6	18.5	> 20.6	14.4	12.5	10.5	tested	
		Avg.	> 20.6 *			> 20.6	17.8	> 20.6	14.0	11.6	10.3		
Extruded			16.2				17.1	15.8	14.0	15.0	14.1	15.0	15.6
Teflon	.0114		16.4				18.3	20.4	16.2	16.8	19.0	16.5	16.5
			19.7				22.0	20.5	16.8	17.0	21.0	17.6	17.2
		Avg.	17.4				19.6	18.9	15.7	16.3	18.0	16.4	16.4
Asbestos			2.0 2.4	3.3	3.1	3.5	1.5	1.0 1.3	2.8	0.8	1.8	0.8	2.6
(Phosphate)	.0054		2.0 2.5	3.5	3.3	3.6	2.6	1.0 1.4	2.9	0.9	1.9	0.9	3.1
			2.0 2.6	3.6	3.4	4.2	3.1	1.0 1.4	3.1	0.9	2.1	1.1	3.2
		Avg.	2.0 2.5	3.5	3.3	3.8	2.5	1.0 1.4	2.9	0.9	1.9	0.9	3.0
Asbestos			2.8 2.4				1.5	1.2	3.3	0.9	3.8	0.7	2.9
ML Coated	.0052		3.2 2.5				1.5	1.4	3.8	1.1	3.9	0.9	3.7
			3.4 2.6				2.5	1.4	4.0	1.1	4.2	1.0	4.8
		Avg.	3.1 2.5				1.8	1.3	3.7	1.0	4.0	0.9	3.8
Glass Fiber			1.3	7.4				2.0					
ML Coated			1.3	8.1		11.2		2.0					
			1.4	8.4		11.3		2.3					
		Avg.	1.3	8.0		11.3		2.1					

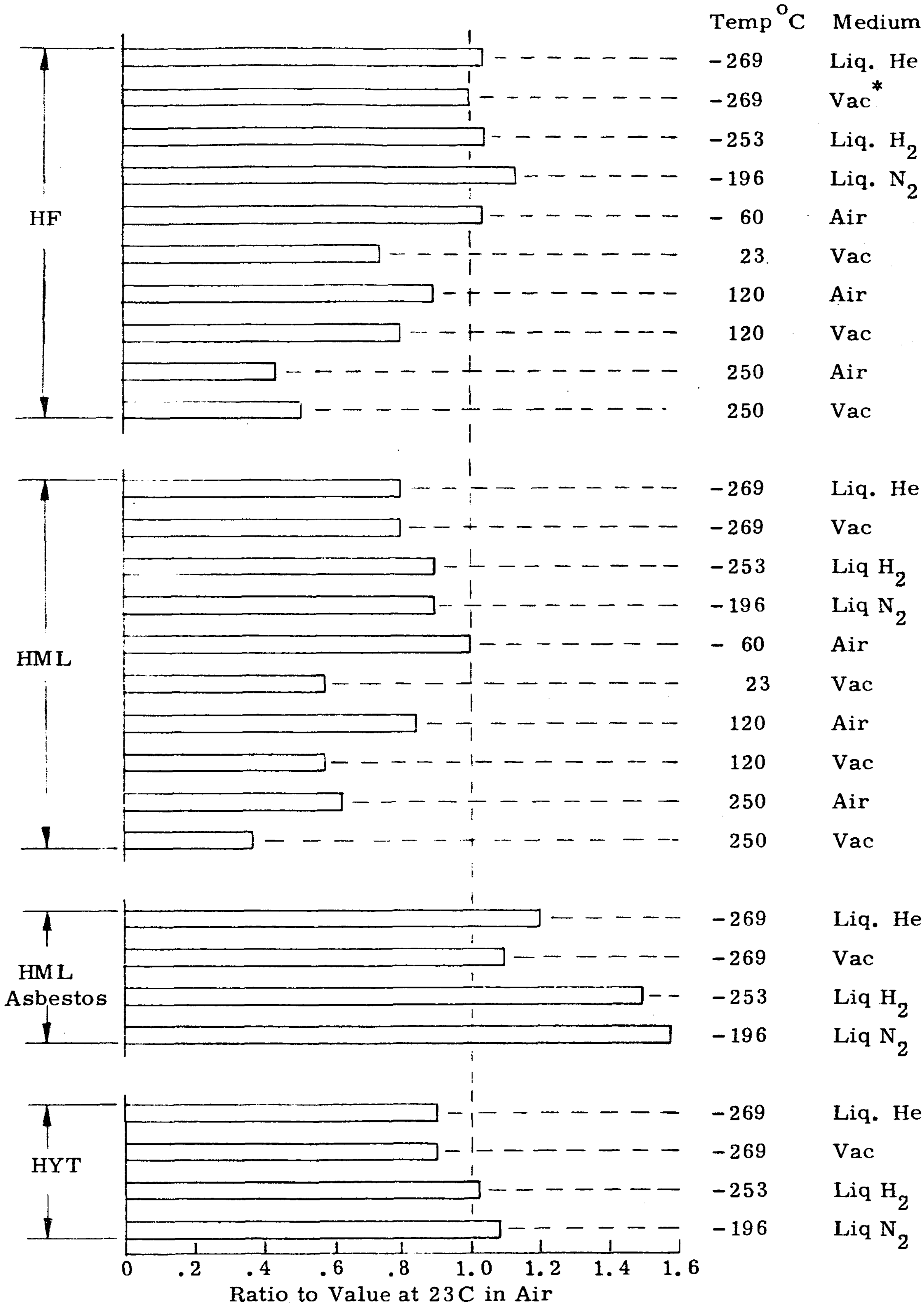
* Maximum test voltage was limited to 20.6 KV



* Vacuum of 10^{-5} Torr or better

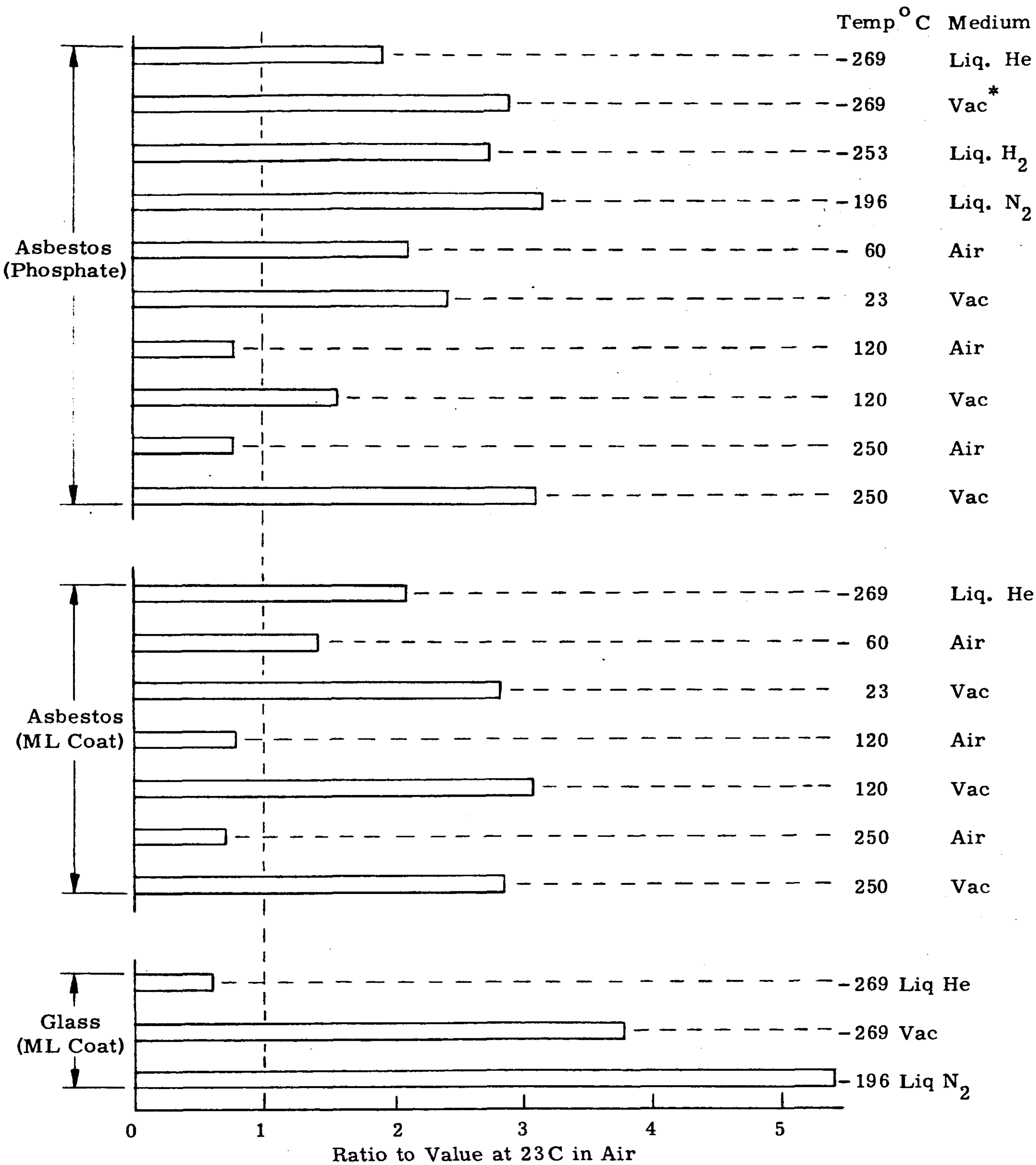
(?) Ratio could not be obtained because Breakdown Voltage was above the test limit of 20.5 KV

Fig. 1 Comparison of Breakdown Voltage under different Test Ambients for Extruded Wires



* Vacuum of 10^{-5} Torr or better

Fig. 2 Comparison of Breakdown Voltage under Different Test Ambients for Film Coated Wires



* Vacuum of 10^{-5} Torr or better

Fig. 3 Comparison of Breakdown Voltage under different Test Ambients for Fibrous Coated Wires electrode area as contrasted to the point contacts of the usual twisted pair specimen. In either case, the voltage breakdown would be reduced. At cryogenic temperatures the asbestos covering has much better dielectric properties and the overall breakdown voltage is significantly increased. The results plotted for HML in Fig. 2 are the averaged results for all of the different types of HML as given in Table IV-A. In consequence, it would appear that the small decrease in breakdown strength for HML at low temperatures, particularly at -269C, is significant. Heavy Formex, HF, does not show a similar decrease at low temperatures and the slight decrease shown for the polyester wire, HYT, may not be significant. Since no mechanical damage is evident in HML specimens exposed at liquid helium temperature, -269C, it is difficult to explain the lowered breakdown voltage even though the decrease is small.

The results at elevated temperatures and in vacuum shown in Fig. 2 are also very interesting. The breakdown voltage of both HF and HML is sharply decreased at elevated temperatures. Except for one anomaly with HF at 200C, the breakdown voltage in vacuum is even lower. To the author's knowledge, no outgassing studies in vacuum have been made with HML but the weight loss over long periods of time at elevated temperatures is known to be very low. It would seem difficult to apply the outgassing hypothesis to explain the relatively low breakdown voltage obtained with HML in vacuum unless perhaps a bit of residual solvent is still being evolved. The decrease in breakdown voltage at elevated temperatures with HML is quite surprising in view of its recognized thermal stability and relatively low dielectric losses at elevated temperatures.

In Fig. 3 the effect of vacuum on the breakdown voltage of fibrous insulations is shown to be just the opposite to that with film coated wires. The very large increases in breakdown voltage in vacuum and in the cryogenic liquids has, in fact, made the use of a different scale necessary. However, it is immediately apparent that the breakdown voltage for wires immersed in liquid helium is relatively low and for the glass fiber served wire even lower than in air at room temperature. It will be remembered from the work of the first year on the subject contract that the breakdown voltage of liquid helium is quite low but higher than in air at room temperature. Therefore, the very low breakdown voltage of glass fiber in liquid helium must be considered particularly as contrasted to the higher breakdown voltage for felted asbestos under the same conditions (see Table IV-B). It is proposed that the "tight" construction of the felted asbestos provides a dielectric "barrier" action in liquid helium much like paper does in oil at normal temperatures. However, if such barrier action is responsible for the higher breakdown voltage of asbestos in liquid helium, it is difficult to see why it does not also function similarly in liquid nitrogen, where, in fact, the breakdown voltage for the glass fiber is much higher than in felted asbestos. During a recent European trip (see Appendix) the author discovered that the electrical properties of liquid helium are in some respects at least unlike other liquids and that much remains to be learned about the dielectric performance of cryogenic liquids.

The performance of both asbestos insulated wires in vacuum at elevated temperatures is quite good as shown in Fig. 3, but reference to Tables IV-A and IV-B will show that even so the actual values of breakdown voltage in vacuum are not in general as high for asbestos as for the film coated wires. It should be remembered, also, that the thickness of the fibrous insulation is several times that of the film coating. Nevertheless, the relatively good breakdown performance of fibrous insulations in vacuum is important and does not seem to be consistent with the outgassing theory for explaining the relatively poor breakdown performance of film coated wires in vacuum (fibrous insulations must outgas at least as much as the film coatings in vacuum).

Breakdown Voltage after Thermal Aging

Breakdown voltages after various periods of thermal aging and also after 15 days at 95% RH - 80C are compared in Table V. After thermal aging, measurements were made in air at 23C and also in liquid helium at -269C. After moisture exposure measurements were made at 95% RH and 80C. Another set of measurements was made after the cabinet containing the samples was carefully cooled to 23C and 95% RH so as to avoid moisture condensation. Finally, a third set of samples was removed quickly from the moisture conditioning chamber and immersed as soon as possible in liquid helium (-269C). Figs. 4 and 5 compare the ratio of average breakdown voltage after aging to the average value before aging, so as to more clearly indicate the effect of thermal aging. Similar results in chart form for moisture exposure are plotted in Fig. 6. In Fig. 4 it is immediately apparent that the breakdown strength of PVC in liquid helium is markedly reduced by thermal aging in vacuum but not in air. Precisely, the opposite effect occurs with HF (see Fig. 5) in that aging at 125C in air but not in vacuum results in decreased dielectric strength. While the thermal degradation of HF in air at 125C is apparent from the decrease of breakdown voltage at both 23C and in liquid helium, the effect in liquid helium is more marked. It is perhaps very surprising that the thermal degradation of PVC after aging at 125C in vacuum is measured by decreased breakdown voltage only at -269C and not at 23C. The apparent difference in the thermal degradation mechanisms for PVC and HF seems more obvious. It is likely that HF in air crosslinks by oxidation to become more brittle. PVC, on the other hand, probably looses plasticyzer and perhaps other components which provide stabilization against autocatalytic degradation from evolved HCl.

In Fig. 4 it is evident that Teflon aged at 250C (and perhaps at 120C) decreases in voltage breakdown measured at room temperature but <u>not</u> when measured in liquid helium. No explanation for such peculiar performance can be advanced. It is unfortunate that thermal aging at 250C was not also performed in vacuum. (PVC and HF were not aged at 250C because they would char completely).

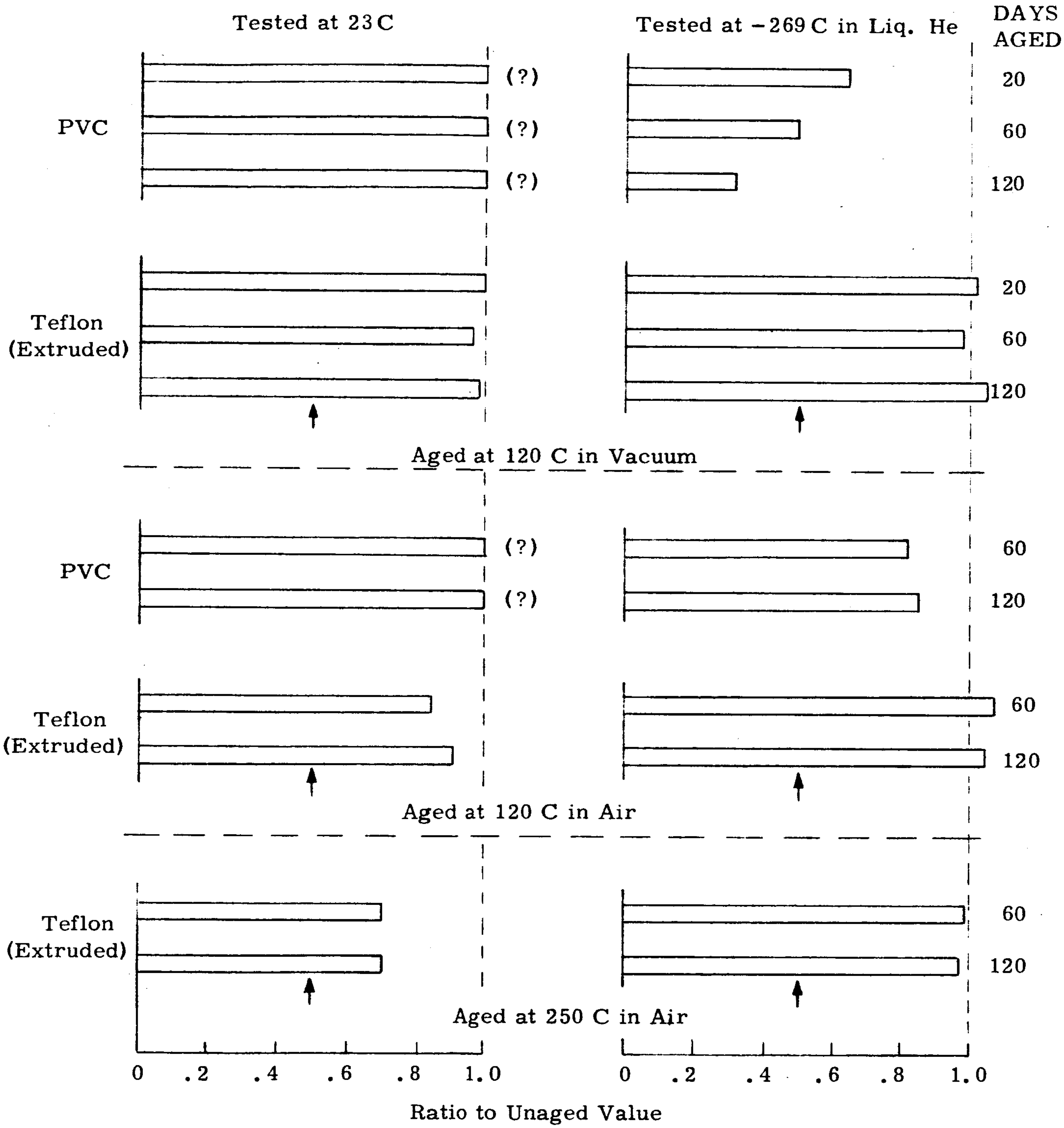
From Fig. 5 it may be observed that the voltage breakdown of HML is not significantly affected by thermal aging even at 250C (it was shown earlier that both high and low temperatures in themselves do decrease the breakdown voltage of ML, however). Such thermal stability for an

Table V

Summary of Thermal and Humidity Aging Effects on the Voltage Breakdown (KV) Of Insulated Wires at Room and Cryogenic Temperatures

Insulated Wire (Temperature of Test) →		Original 25C	Original 4K	Thermal Aging: Vacuum at 120 C, 20 (Days) 25C	20 (Days) 4K	60 Days 25C	60 Days 4K	120 Days 25C	120 Days 4K	Air at 120 C, 60 Days 25C	60 Days 4K	120 Days 25C	120 Days 4K	Air at 250 C, 60 Days 25C	60 Days 4K	120 Days 25C	120 Days 4K	Moisture Aging for 15 Days (95% Relative Humidity at 80 C), Tested at: 25C	4K	95% R.H., 80 C
Heavy Formex	Avg.	7.1	8.0	8.5	7.6	7.8	7.8	7.1	7.5	5.4	5.6	5.7	4.5	(Charred off at 250 C)				3.3	5.9	3.1
(0.0013" Wall)	Min.	6.1	7.9	8.4	6.7	7.8	6.8	6.2	6.8	4.6	4.6	4.9	3.4					3.1	4.2	1.9
	Max.	8.8	8.1	8.6	8.3	7.8	8.9	8.4	8.2	6.8	6.7	6.7	5.3					3.4	7.6	4.3
Heavy ML (G.E.)	Avg.	13.3	9.0	16.6	9.8	15.9	8.4	15.6	8.9	14.2	10.1	13.1	7.6	14.1	8.5	13.6	8.3	14.3	7.3	10.8
(0.0014" Wall)	Min.	11.4	8.7	15.6	9.7	14.0	8.4	11.6	8.8	12.5	9.4	10.2	7.6	10.4	8.3	11.6	8.1	14.0	5.0	9.5
	Max.	14.6	9.4	17.4	9.8	17.8		17.6	9.0	15.6	10.6	15.6		17.2	8.6	14.6	8.6	14.6	9.5	11.6
Heavy ML (Phelps-Dodge)	Avg.	10.5	7.2	10.9	8.7	11.5	6.8	11.9	5.8	10.1	7.4	10.4	7.2	8.7	7.4	10.0	7.6	9.0	8.0	8.5
(0.0011" Wall)	Min.	9.1	5.0	9.3	8.3	11.3	4.7	10.2	4.4	8.0	7.3	9.5	6.8	8.1	6.8	9.7	7.6	7.4	8.0	7.8
	Max.	11.7	9.6	12.2	9.0	11.6	7.8	13.2	7.2	12.1	7.5	12.0	8.8	9.3	7.8	10.3	7.7	10.2		9.1
Teflon (Extruded)	Avg.	18.9	17.4	18.7	17.7	17.1	18.5	18.[illegible]	19.5	15.8	19.6	17.4	19.3	13.1	17.2	13.1	17.0	19.9	17.4	
(0.0114" Wall)	Min.	15.8	16.2	17.4	15.8	15.6	14.6	15.6	18.6	12.6	18.8	17.0	17.8	12.8	16.2	11.6	13.4	19.4	17.4	
	Max.	20.4	19.7	19.4	19.8	18.8	20.5	20.4	20.5	19.2	20.5	18.2	20.5	13.4	18.5	15.4	19.4	20.3		
Poly Vinyl Chloride	Avg.	>20.5	>20.5	>20.5	12.9	>20.5	10.6	>20.5	6.5	>20.5	16.9	>20.5	17.7	(Not aged at 250 C)				18.0	16.6	
(0.0071" Wall)	Min.				10.2		5.0		4.2		16.2		17.4					16.6	16.6	
	Max.				14.6		13.8		8.2		17.6		18.4					20.5		
Asbestos (Phosphate)	Avg.	1.4	2.5	1.0	2.1	1.0	1.9	1.0	1.8	1.1	1.8	1.0	1.8	1.0	1.9	1.0	2.1	1.0	2.3	
(0.0054" Wall)	Min.	1.4	2.4	1.0	2.0	0.7	1.7	0.9	1.6	1.1	1.6	1.0	1.7	0.9	1.7	0.9	2.0	1.0	2.2	
	Max.	1.5	2.6		2.3	1.1	2.1	1.1	1.9		2.0		1.9	1.1	2.0	1.0	2.2		2.3	
Asbestos (ML Coated)	Avg.	1.3	2.5	1.1	2.4	1.2	2.4	1.1	2.6	1.1	2.3	1.1	2.4	1.2	2.4	1.1	2.4	1.4	2.4	0.5
(0.0064" Wall)	Min.	1.2	2.4	1.0	2.3	1.1	2.3	1.1	2.5	1.0	2.2	1.0	2.3	1.1	2.3	1.0	2.3	1.4	2.3	0.5
	Max.	1.4	2.6	1.2	2.4	1.3	2.5		2.6	1.2	2.6	1.1	2.6	1.3	2.6	1.2	2.4		2.4	
Triple ML (Phelps-Dodge)	Avg.	13.6	11.2																	6.1
(0.0016" Wall)	Min.	11.2	10.8																	5.4
	Max.	15.1	11.5																	7.1

Test Conditions: Average of 3 Breakdowns (20 KV/Minute Rise, 60 Cycles) of Twisted Pairs of Wire in Room Temperature Air or Under Liquid Helium at 4K.



(?) Ratio could not be obtained because Breakdown Voltage was above the test limit of 20.5 KV.

* For most of the aging period a vacuum of about 10^{-5} Torr was held.

Fig. 4 Comparison of Breakdown Voltage after Thermal Aging for Extruded Wires

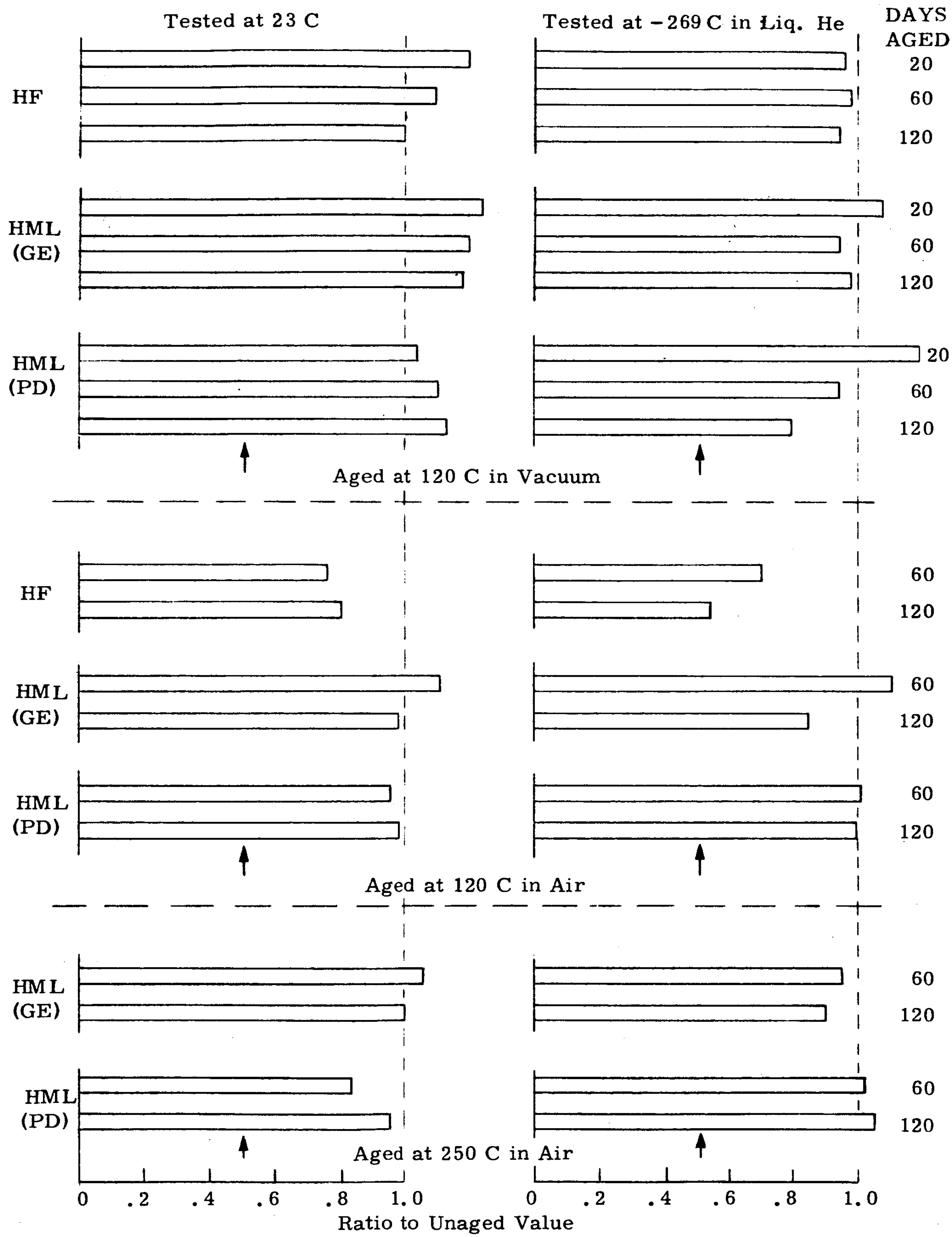


Fig. 5 Comparison of Breakdown Voltage after Thermal Aging for Film Coated Wires

organic material is remarkable. The voltage breakdown of the asbestos insulated wires was also not significantly affected by thermal aging as shown in Table V (a figure was not made) but such performance is to be expected of inorganic materials.

The effect of moisture exposure is graphically shown in Fig. 6. Some surprising and confusing results appear. For example, the breakdown voltage of ML coated asbestos does not seem to be adversely affected by moisture exposure. In view of the recognized moisture absorption by asbestos such a result can hardly be believed. However, the voltage breakdown of aluminum phosphate impregnated asbestos is degraded by moisture exposure as would be expected. The increased breakdown voltage when wet asbestos insulation is tested at -269C is interesting but expected on the basis of results obtained previously. In liquid helium the HML(GE) exhibits decreased breakdown voltage but precisely the opposite effect occurs with HML(PD). It is tempting to theorize that decreased breakdown voltage after moisture exposure, when measured at room temperature or at 80C, is due to absorbed moisture but that a similar decrease when measured in liquid nitrogen may denote a more permanent moisture degradation such as hydrolytic, molecular scission. Such a hypothesis would mean that HML(PD) is more resistant to hydrolytic molecular scission than HML(GE). The information to date is inadequate for such a sweeping conclusion but the subject will be re-examined later in reference to flexibility measurements.

Dissipation Factor after Thermal Aging

In the Sixth Quarterly Report, January 1963, the effect of thermal aging on the capacitance of various wire insulations has been compared when measured both at room temperature and at -269C in liquid helium. The changes are small for the extruded and film coated wires. For the asbestos insulation large changes in capacitance occur between room temperature and -269C probably because the ever present moisture in asbestos freezes and at very low temperatures ionic conductivity does not contribute appreciably to dielectric losses. However, capacitance is not a sensitive indicator of changes produced by thermal aging.

Dissipation factor does provide a sensitive indication of change brought about by thermal aging. A complete tabulation of results is given in Table VI. To make comparison easier, average results as ratios are shown in Figs. 7A and 7B. These two figures compare 1 KC measurements made at 23C-50% RH. Measurements made in liquid nitrogen and in liquid helium are reported also in Table VI but do not provide a particularly useful means of studying aging effects.

It is particularly interesting to compare test results after aging at 120C in vacuum (shown on the top half of Fig. 7A) to aging at 120C in air (shown in the bottom half of Fig. 7A). It is apparent, immediately, that the dissipation factor of PVC increases slightly when aged in vacuum and decreases when aged in air. In contrast, the dissipation factor of HF increased only when aged in air. These results correlate

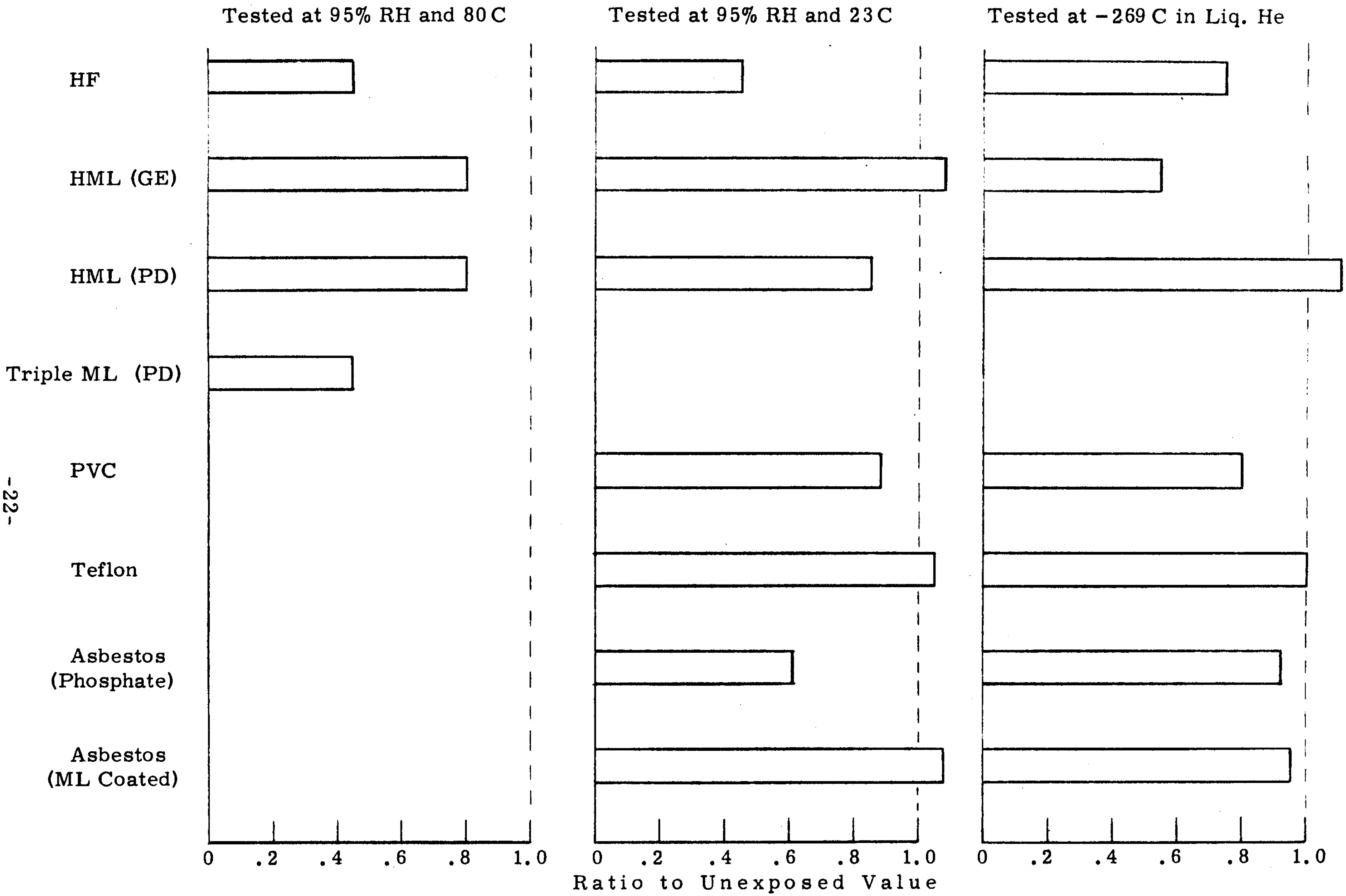


Fig. 6 Comparison of Breakdown Voltage after Exposure for 15 Days at 95% RH and 80 C

Table VI

Effect of Thermal Aging on Dissipation Factor at 1 KC

Wire	At 23 C Before Aging	At 23 C After 60 days 120 C/Air	At 4.2 K After 60 d 120 C/Air	At 23 C After 120 d 120 C/Air	At 77°K After 120 d 120 C/Air	At 4.2 K After 120 d 120 C/Air	At 23 C After 60 d 120 C/Vac	At 4.2 K After 60 d 120 C/Vac	At 23 C After 120 d 120 C/Vac	At 77°K After 120 d 120 C/Vac	At 4.2 K After 120 d 120 C/Vac	At 23 C After 60 d 250 C/Air	At 4.2 K After 60 d 250 C/Air	At 23 C After 120 d 250 C/Air	At 77°K After 120d 250 C/Air	At 4.2 K After 120 d 250 C/Air
Teflon	.00035	.00024	.00000(OK)													
	.00057	.00034	.00000(OK)													
"	.00034			.0000(?)	<.00000	.00000										
	.00082			.00062		.00000										
"	.00087						.0006	.00007								
	.00107						.0008	.00015								
"	.00086								.00060	.00000(?)	.00026(?)					
	.00088								.00056	.00000(?)	.00124(?)					
"	.00013													.00026	.00000	.00000
	.00014													.00032	.00000	.00000
PVC	.062	.0622	.00000													
	.100	.0664	.00000													
"	.099			.0408	.0008											
	.062			.0429	.0007											
"	.060						.0702	.00000								
	.059						.0678	.00000								
"	.057								.0749	.00096						
	.058								.0709	.00092						
Heavy	.0064	.00974	.00000													
Formex	.0058	.00877	.00002													
"	.0057			.0094	.0014	.0000(?)										
	.0059			.0106	.0013	.000107										
"	.0057						.00539	.00012								
	.0058						.00606	.00000								
"	.0063								.0057	.0011	.00027					
	.0063								.0057	.0011	.00021					
HML	.00097	.00087	.00000													
(G.E.)	.00096	.00089	.00000													
	.00116			.00097	.00080	.00000										
"	.00147			.00089	.00080	.00000										
	.00088						.00076	.00000								
"	.00115						.00097	.00000								
	.00121								.0011	.00070	.0003					
"	.00114								.00093	.00076	.0006					
	.00092											.00165	.00000			
"	.00083											.00117	.00000			
	.00076													.0029	.0004	.00000
"	.0011													.0036	.0005	.00005
HML (PD)	.0013	.00096	.00000													
	.0016	.0011	.00000													
"	.0014			.00068	.00070	.00000										
	.0012			.00070	.00068	.00000										
"	.0021						.00132	.00000								
	.0018						.0012	.00000								
"	.0019								.00063	.00059	.00000					
	.0023								.00115	.00074	.00004					
"	.0015													.0021	.0012	.00000
	.0018													.0144	.0011	.00000

Table VI (Contd.)

Effect of Thermal Aging on Dissipation Factor at 1 KC

Wire	At 23 C Before Aging	At 23 C After 60 days 120 C/Air	At 4.2 K After 60 d 120 C/Air	At 23 C After 120 d 120 C/Air	At 77°K After 120 d 120 C/Air	At 4.2 K After 120 d 120 C/Air	At 23 C After 60 d 120 C/vac	At 4.2 K After 60 d 120 C/Vac	At 23 C After 120 d 120 C/Vac	At 77°K After 120 d 120 C/Vac	At 4.2 K After 120 d 120 C/Vac	At 23 C After 60 d 250 C/Air	At 4.2 K After 60 d 250 C/Air	At 23 C After 120 d 250 C/Air	At 77°K After 120 d 250 C/Air	At 4.2 K After 120 d 250 C/Air
Inorganic	0.377	0.2055	.00000													
Bonded	0.322	0.1835	.00000													
Felted	0.419			0.1598	.0005	.00000										
Asbestos	0.312			0.160	.0005	.00000										
	0.372						0.169	.00000								
	0.323						0.156	.00000								
	0.380								0.148	.0004	.00007					
	0.358								0.159	.0003	.00021					
	0.452											0.219	.00000			
	0.486											0.250	.00000			
	0.344													0.215	.0002	.00003
	0.363													0.220	.0002	.00008
ML Coated	0.26	0.243	.00000													
Felted	0.22	0.211	.00000													
Asbestos	0.26						0.275	.00000								
	0.20						0.218	.00000								

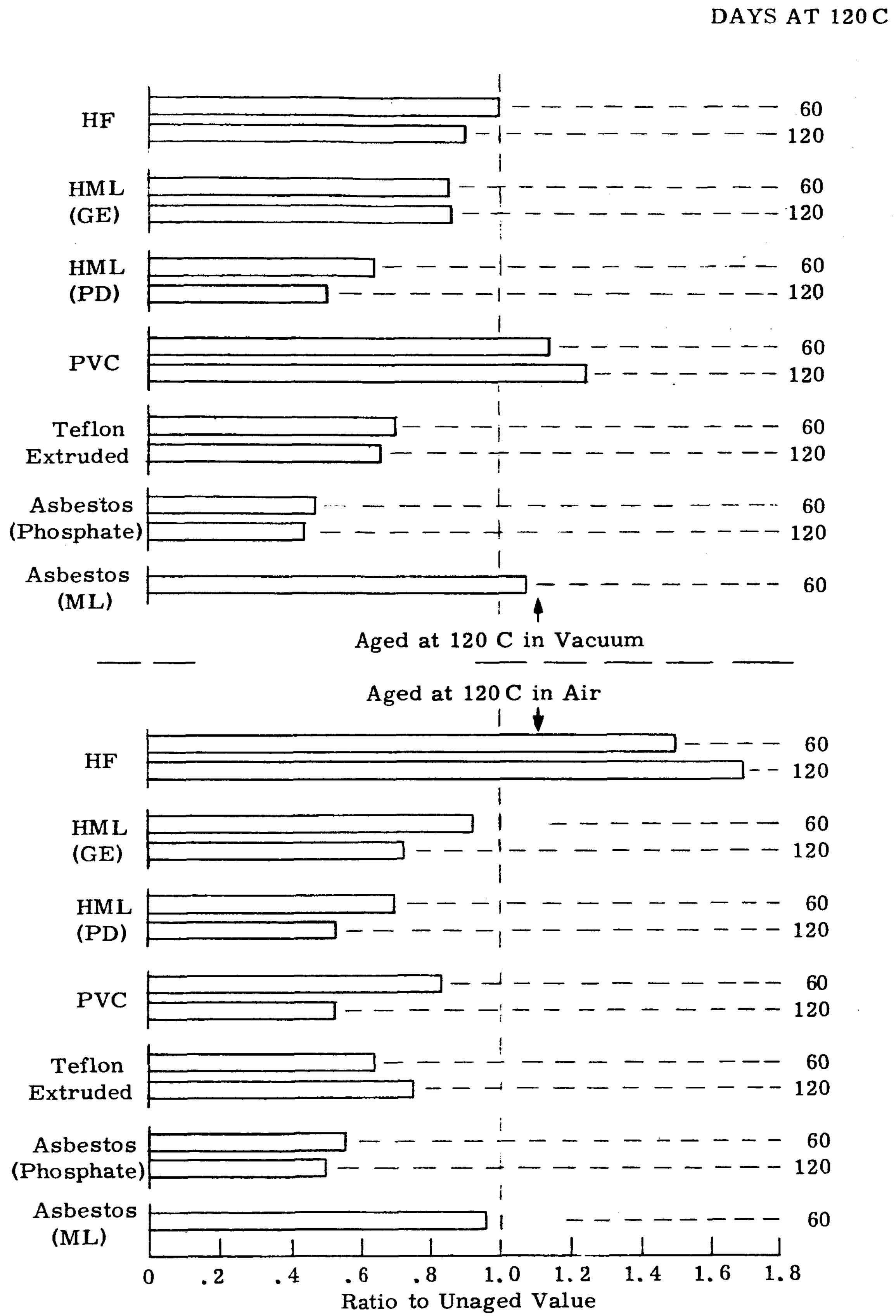
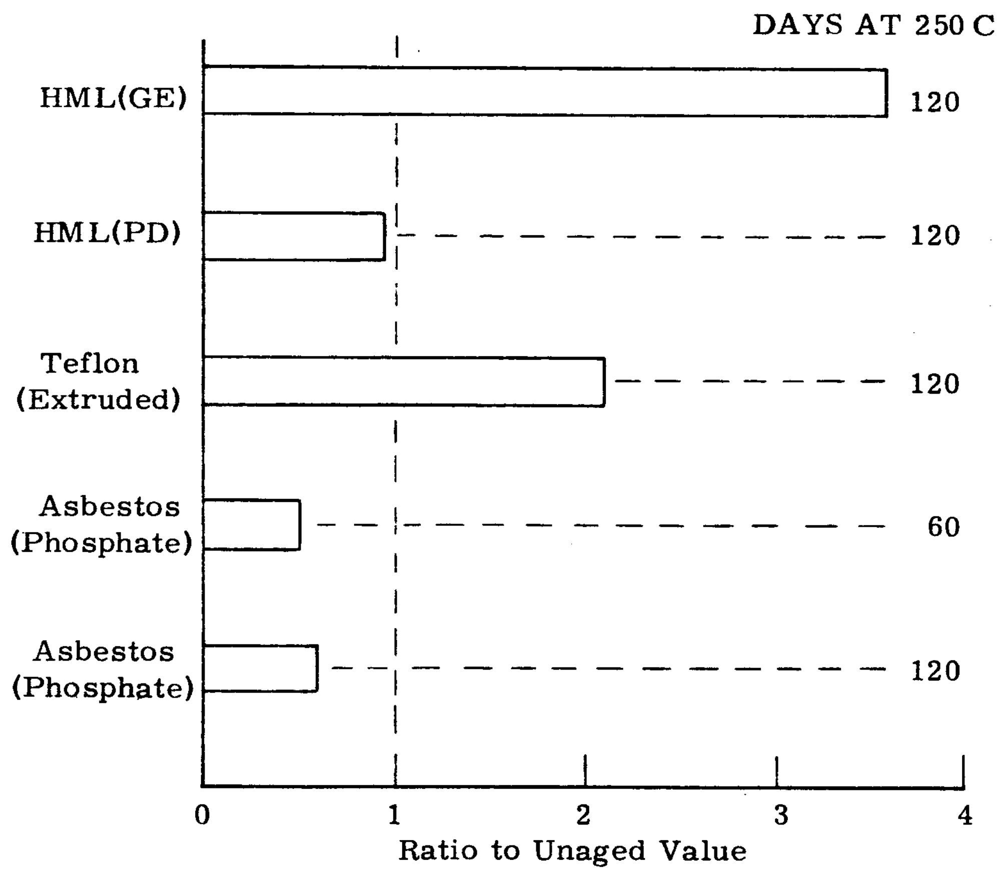


Fig. 7A Comparison of Dissipation Factor after Thermal Aging at 120 C

Note: 1 Kc Measurements made at 23 C - 50% RH



Note: 1 Kc measurements made at 23 C - 50% RH

Fig. 7B Comparison of Dissipation Factors after Thermal Aging at 250 C with the results of breakdown tests described earlier. They indicate that thermal degradation in PVC is accelerated by loss of plasticyzer in vacuum but that degradation in Formex results from oxidation.

The decrease in dissipation factor with ML samples and also for Teflon at 120C in both air and vacuum is interesting. The decrease for ML can perhaps be ascribed to cure but the change in Teflon is much more difficult to understand. It is possible that some of the high molecular weight, hydrocarbon lubricant (usually cetane) used in the extrusion of the Teflon has been trapped in the fusion process and is removed during thermal aging. The decrease in dissipation factor for aluminum phosphate impregnated asbestos after aging at 120C may be attributed to loss of moisture in the impregnant since ML coated asbestos does not show the same decrease.

The results in Fig. 7B show results after aging at 250C opposite to those which occur after aging at 125C (except for asbestos). The relatively large increase in dissipation factor after aging at 250C in air for HML(GE) and for Teflon may be due to the development of an oxide layer on the copper conductor which introduces electrical losses. If copper oxidation alone is responsible, it is difficult to explain the difference in performance between the General Electric and Phelps Dodge ML coatings. It may be that the PD ML coating adheres better to the copper and thereby with it the oxide growth is inhibited.

Capacitance and Dissipation Factor after Moisture Exposure

Capacitance and dissipation factor measurements after moisture exposure are reported in Tables VII and VIII for all of the principal wires except asbestos. (The values of dissipation factor for asbestos after moisture exposure are too high to be easily measured.) At low temperatures (Condition 3), the capacitance changes shown in Table VII are due to change in temperature rather than to the effect of moisture exposure. The changes in capacitance as shown by the ratio figures for measurements both at 95% RH and after drying are relatively small. The changes in dissipation factor are larger (see Table VIII) and have been plotted as ratios in Fig. 8*. In view of the effect of moisture in decreasing the voltage breakdown of HF, it is surprising that no change in dissipation factor is indicated after moisture exposure. The increase in dissipation factor for HML specimens after drying at room temperature is very difficult to explain and the increase for Teflon under the same condition is the most puzzling of all. The dissipation factor values for the Teflon specimens remained high even when dried at 80^{o}C for 24 hours. At this point, it should be remembered that the breakdown voltage of HML is always decreased at -269C (in vacuum or in liquid helium) as compared to room temperature. Perhaps very cold temperatures cause some sort of deterioration in HML coatings which has not been characterized so far. It should be noted, also, that other investigators have sometimes found strange types of degradation in electrical properties when Teflon has been exposed to moisture.

* The values of dissipation factor at -296C in liquid helium have not been included. Very low values were obtained which do not show significant effects of moisture exposure.

Table VII

Capacitance Before and After Moisture Exposure

Spec.	Sample No.	Test Condition 1	2	*	3	4	*
PVC	1	39.81	39.17	.985	25.53	39.28	.99
	2	41.47	40.61	.98	27.16	41.16	1.01
	3	42.91	41.40	.965	27.83	40.66	.95
				.98			.98
HF	1	65.31	71.64	1.10	65.09	67.94	1.04
	2	62.61	70.88	1.13	63.82	67.54	1.08
	3	60.57	67.78	1.10	61.80	64.95	1.03
				1.11			1.05
Teflon	1	14.63	13.79	.945	14.57	13.78	.94
	2	14.55	13.79	.945	14.89	14.13	.97
	3	13.73	12.59	.92	14.03	12.95	.945
				.94			.95
HML/GE	1	59.17	57.77	.975	64.33	65.57	1.085
	2	65.49	67.22	1.03	71.90	72.14	1.075
				1.05			1.08
HML/PD	1	67.39	69.77	1.03	77.30	73.63	1.09
	2	68.09	70.33	1.03	76.44	68.03	1.00
	3	67.30	68.25	1.02	74.25	73.10	1.085
				1.03			1.06

* Ratio to Unexposed Value

Condition 1 -- As received - 23C & 50%RH

" 2 -- After exposure at 80C-95% for 15 days
Specimen removed from chamber and placed immediately in chamber at 23C 95% RH and measured after 36 hrs. at 23C-95%RH

" 3 -- Removed from condition 2 above and plunged directly into liquid nitrogen before measurement

" 4 -- Removed from Condition 3 above and kept at 23C 50% RH for 72 hrs. before measurement.

Table VIII

Dissipation Factor Before and After Moisture Exposure

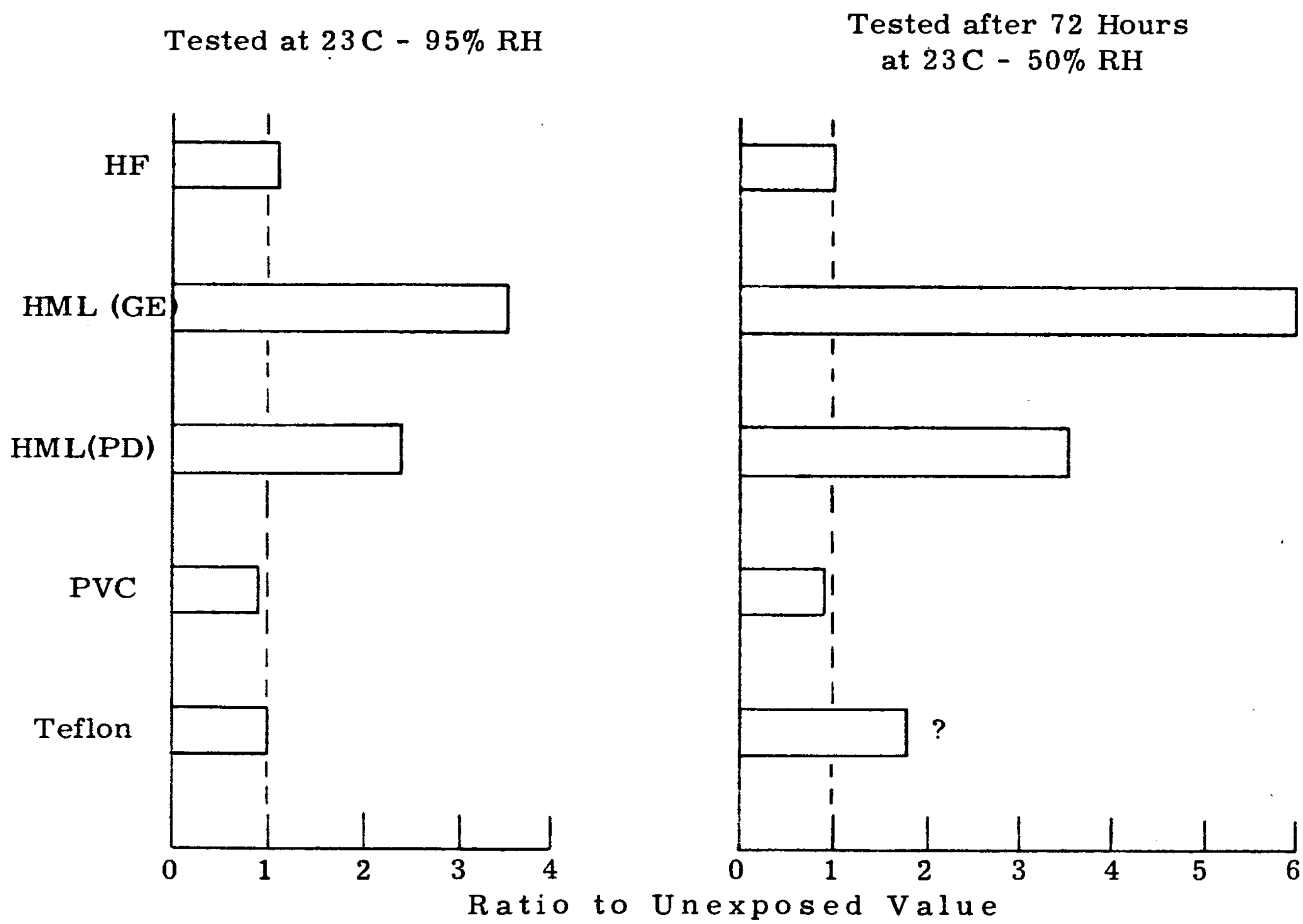
Spec.	Sample No.	Test Condition 1	2	3	4
PVC	1	0.0975	0.091	0.0001	0.0933
	2	.0958	.090	.0007	.0897
	3	.0947	.088	.0002	.0909
HF	1	0.00717	0.0071	0.0006	0.0062
	2	.00580	.0070	.0012	.0066
	3	.00590	.0068	.0009	.0059
Teflon	1	0.00077	0.00057	< 0.0000	0.0006
	2	.00078	.0012	< .0000	.0025
	3	.00069	.00050	.0009	.0024
HML/GE	1	0.00164	0.0054	0.0001	0.0098
	2	.00159	.0059	.0008	.0095
HML/PD	1	0.00120	0.0030	0.0003	0.0057
	2	.00114	.0024	.0002	.0019
	3	.00117	.0029	.0006	.0054

Condition 1 -- As received - 23C & 50% RH

" 2 -- After exposure at 80C-95% for 15 days
Specimen removed from chamber and placed immediately in chamber at 23C 95% RH and measured after 36 hrs. at 23C- 95% RH

" 3 -- Removed from Condition 2 above and plunged directly into liquid nitrogen before measurement

" 4 -- Removed from Condition 3 above and kept at 23C 50% RH for 72 hrs. before measurement.



Note: Measurement made at 1 Kc

Fig. 8 Comparison of Dissipation Factors after Exposure for 15 Days at 80 C and 95% RH

Surface and Volume Resistance

The volume resistance of most good insulating materials is difficult to measure, particular on short lengths of wire. On long lengths of wire, volume resistance measurements are useful in determining the presence or effect of foreign inclusions, holes or discontinuities. These defects, however, usually prevent determination of the intrinsic resistance properties. However, at high humidities volume and resistance can more easily be measured.

Surface resistance usually can be measured at 50% RH because even such low humidities have an effect on the surface condition. Some problems are involved with surface resistance measurements of insulation on wire particularly at high humidity when lowered volume resistance may interfere (the conductor is usually connected to the guard circuit and too low a resistance to guard will upset such measurements). When resistance measurements at very low temperatures are needed, electrode problems become particularly important. The bundled, concentric wire sample developed in this program for the measurement of AC characteristics has been found not suitable for DC volume measurements and is inapplicable for surface measurements. Silver paint or exaporated gold electrodes are not suitable for the asbestos samples and both flake off other insulations even at liquid nitrogen temperatures as described in previous reports. Consequently, for the subject work, thin lead foil electrodes were used. Two 3/8 inch wide lead foil electrodes were wound tightly around the insulated wire and spaced 1/8 inch apart. Surface resistance is measured between the two electrodes with the conductor connected to guard so as to avoid as far as possible the errors introduced by volume resistance. Volume resistance, in turn, is measured by connecting the two surface electrodes together and measuring to the center conductor without attempting to use guarding techniques which would be complicated and probably ineffective in preventing errors introduced by low values of surface resistance. The somewhat unreliable contact provided by foil electrodes against the insulation surface may have a marked effect on volume resistance but probably much less effect on surface resistance. Overall, resistance measurements at best provide, primarily, an indication of change and can be most effectively used to determine the effect of exposure conditions such as high humidity. In consequence, no attempt has been made to calculate resistivity since only relative values are needed.

Values of both volume and surface resistance are shown in Table IX. A comparison can be made of the surface resistance at both 50 and 95% RH. Except for asbestos, the surface resistivity for the different wire insulations is remarkably similar and, of course, lower by about three decades at 95% RH as compared to 50% RH. When the wet samples were plunged into liquid nitrogen, the surface resistance was higher than at 50% RH and 23C. The relatively high surface resistance for asbestos in liquid nitrogen is expected on the basis of previous work.

The very low volume resistance of the asbestos insulation after moisture exposure is expected and the five decade increase after

Table IX

Average Values of Surface and Volume Resistance
Before and After Moisture Exposure

	Surface Resistance	Surface Resistance after 15 Days at 80C-95% RH		Volume Resistance after 15 days at 23C-95% RH	
	at 23C-50% RH	at 23C-95% RH	In Liq. N_2	at 23C-95% RH	In Liq. N_2
HF	2.2×10^{15}	6.6×10^{13}	1.6×10^{16}	1.6×10^{12}	1.4×10^{12}(?)
HML(GE)	1.7×10^{16}	2.2×10^{13}	2.7×10^{16}	6.5×10^{12}	1.1×10^{14}
HML(PD)	1.2×10^{16}	1.7×10^{13}	8.3×10^{15}	1.2×10^{12}	2.1×10^{14}
PVC	5.3×10^{15}	3.0×10^{13}	1.9×10^{16}	2.6×10^{12}	9.2×10^{10}(?)
Teflon (Extruded)	8.6×10^{15}	1.2×10^{13}	1.5×10^{16}	1.9×10^{13}	2.5×10^{12}(?)
Asbestos (Phosphate)	2.3×10^{11}	*	1.1×10^{15}	1.5×10^{5}	1.9×10^{10}

* Leakage to guard electrode (center wire) prevented measurement

(?) Unexplained low values -- see text

NOTES: Except for asbestos, volume resistance at 50% RH lies above the sensitivity limits of the instruments used; i.e., $> 10^{17}$

The test specimen consisted of a single wire with two 3/8" wide, tightly wrapped, lead foil electrodes spaced 1/4 inch apart. For surface measurements the wire itself was connected to guard. A test voltage - 500 volts (except 10 or 25 volts with asbestos) - was applied for 1 minute before measurements were made.

immersion in liquid nitrogen is not surprising. However, the volume resistances for the other wires after moisture exposure are lower than might be expected, particularly for PVC and Teflon. The decreased volume resistance for three of the wire insulations (indicated in Table IX by (?)) after immersion in liquid nitrogen is amazing! However, the odd performance of ML in other tests at liquid nitrogen temperature is curiously not repeated in the case of volume resistance which increases as might be expected but certainly even so not to the high values for dry materials at room temperature. It is conceivable, but not probable, that the metal foil electrodes grip the test specimens more lightly in liquid nitrogen than they do at room temperature. More likely some uncharacterized effect of absorbed moisture (ice) at very low temperatures is involved and much more study is needed to understand the phenomenon.

Repeated Mandrel Flexibility as a Function of Temperature

Repeated mandrel flexibility at low temperatures has been used throughout the subject program to determine mechanical performance of wire insulations at very cold temperatures. Very little difference in flexibility has been noted in tests at -296C in liquid helium and at -196C in liquid nitrogen. At both of these temperatures, all of the wire insulations except the asbestos are much less flexible than at room temperature. Obviously, at some intermediate temperature, intermediate values for flexibility will be obtained and different materials might, in fact, be rated in a different order than at very low temperatures. Consequently, the contract requirement for tests at -60C was welcomed. The -60C temperature is about the lowest that is likely to be encountered under normal conditions at the earth's surface.

Table X provides a comparison of repeated mandrel flexibility at four temperatures for the principal wire insulations of this program. While the order of rating for the different insulations at low temperatures does not change at -60C as compared to the lower temperatures, it is apparent that Teflon and the PVC are no longer brittle at -60C. The performance of the PVC in particular is better than might be expected even though a formulation designed for low temperature use had been selected.

Repeated Mandrel Flexibility after Thermal Aging and Moisture Exposure

Mandrel flexibility tests made at room temperature have been used for many years to follow the deterioration which occurs during aging at elevated temperatures. Table XI gives results of repeated mandrel flexibility at room temperature after chemical aging in vacuum and air at 120C and in air at 250C. HF exhibits no measurable decrease in flexibility after aging in vacuum at 120C for 120 days but failure on a 1/4 inch mandrel was obtained after aging in air at 120C for 60 days. The HML samples were able to withstand a 1 X bend (own diameter) without failure after aging at 120C in both air and vacuum. However, after aging for 60 days at 120C in air at 250C, failure occurred after reverse flexing around a 1/8 inch mandrel and the flexibility decreased

Table X

Repeated Mandrel Flexibility
As a Function of Temperature
(Unaged Samples)

Wire		-269C	-196C	-60C	23C
HF	Failure	1/2	1/2		
	Crazed	1/2 - 1-3/4	1/2 - 1-1/2	1/8 - 1	
	Satisfactory	1-1/4	1	1/8	OK - 1X*
HML(GE)	Satisfactory	OK - 1/8*	OK - 1/8	OK - 1/8	OK - 1X
HML(PD)	Satisfactory	OK - 1/8	OK - 1/8	---	OK - 1X
Triple ML(PD)	Satisfactory	OK - 1/8	OK - 1/8	---	OK - 1X
Teflon (Extruded)	Failure	1	1		
	Satisfactory	1-1/4	1-1/4	OK - 1/8	OK - 1X
PVC	Failure	1-3/4	1-3/4		
	Satisfactory	---	---	OK - 1/8	OK - 1X
Asbestos (Phosphate)	Failure	1/2	1/2		3/4
	Cracks	1/8 - 1/2	1/8 - 1/2	---	1/8 - 3/4
	Satisfactory	3/4	3/4		1
Asbestos (ML Coat)	Failure	1/4	1/8		1/8
	Cracks	1/8 - 1/4	1/8 - 1/4	---	1/8 - 3/4
	Satisfactory	1/2	1/2		1

* OK - 1X indicates no failure when wrapped around its own diameter.

* OK - 1/8 indicates no failure when reverse flexed 10 times around a 1/8" mandrel.

"Failure" involves loss of adhesion with film coatings or a crack through to the copper with fibrous coatings.

Table XI

Repeated Mandrel Flexibility Test at 25°C

(Test Conditions: 10 Reverse Bends Around Mandrel Diameter Shown)

Insulated Wire	Overall Wire Dia. - In.	Pre-Conditioning Before Test: 120 C/Vac 60 Days	120 C/Vac 120 Days	120 C/Air 60 Days	120 C/Air 120 Days	250 C/Air 60 Days	250 C/Air 120 Days	80 C/95% R.H. Air 15 Days
Teflon (Extruded) (0.0114" Wall)	Satisfactory	OK - 1X	OK - 1X*		OK - 1X	OK - 1X	OK - 1X	OK - 1X
Polyvinyl Chloride (0.0071" Wall)	Satisfactory	OK - 1X	OK - 1X	OK - 1X	OK - 1X	No test	No test	OK - 1X
Heavy Formex (0.0013" Wall)	Failure	None	None	1/4	1/4	No test	No test	None
	Satisfactory	OK - 1X	OK - 1X	1/2	3/4		No test	OK - 1X
HML (G.E. Co.) (0.0014" Wall)	Failure		None		None	1/8	1/4	None
	Welts		---		---	1/2 - 3/4	1/2 - 3/4	---
	Satisfactory	OK - 1X	OK - 1X	OK - 1X	OK - 1X		1	OK - 1X
HML (Phelps-Dodge) (0.0011" Wall)	Failure		None		None	1/8	1/2	None
	Welts		---		---	1/2 - 3/4	3/4 - 1	---
	Satisfactory	OK - 1X	OK - 1X		OK - 1X		---	OK - 1X
Asbestos (Phosphate) (0.0054" Wall)	Failure	Not	3/4	Not	3/4	Not	1	1
	Cracks	tested	1/8 - 3/4	tested	1/8 - 3/4	tested	1/8 - 1	3/4 - 1
	Satisfactory		1		1		---	---
Asbestos (ML over-coated) (0.0064" Wall)	Failure	Not	1/8	Not	1/8	Not	1/4	1/4
	Cracks	tested	1/8 - 3/4	Tested	1/4 - 1/2	Tested	1/8 - 1/4	1/8 - 1/2
	Satisfactory		1		3/4		1/2	3/4

* OK - 1X, the wire can be wrapped on its own diameter without visible damage.

"Failure" involves loss of adhesion with film coatings or a crack through to the copper with fibrous coatings.

still further after 120 days at 250C. These tests after thermal aging correlate well with the changes in electrical properties described earlier in this report. However, for mandrel flexibility tests made at room temperature, no decrease in flexibility after thermal aging was noted for the other wires. It is interesting that room temperature flexibility with PVC aged at 120C in vacuum did not show evidence of degradation like that indicated by the electrical tests. However, a number - but not all - specimens of PVC insulated wire aged for 60 days in vacuum at 120C and then bent around their own diameters did split when immersed in liquid helium. Such failures did not occur with PVC even after 120 days aging in air at 120C. The insulated Teflon specimens could also be bent around their own diameter and then be immersed in liquid helium without failure even after aging for 120 days at 250C.

Table XI (in the column at the right) shows that moisture exposure does not adversely affect the room temperature flexibility.

Flexibility measurements were made also at -269C in liquid helium after thermal aging and moisture exposure with the results shown in Table XII. The results after thermal aging are plotted in Fig. 9. It is apparent that the same types of changes which were indicated with flexibility measurements made at room temperature are also apparent for flexibility measurements made at low temperatures. The flexibility tests are, in fact, even more sensitive to changes in thermal aging than the tests made at room temperature except that PVC is so brittle in liquid helium that even unaged samples cannot be evaluated. It is interesting that thermal aging has no significant effect on the flexibility of Teflon after aging even at 250C for 120 days. Thus, correlation is not established for Teflon with changes in electrical properties which were observed and reported earlier.

Results for repeated mandrel flexibility tests in liquid helium after 15 days exposure at 80C and 95% RH are given also in Table XII and shown graphically in Fig. 10. While the room temperature tests gave no indication of degradation from the moisture exposure, the flexibility tests in liquid helium indicate marked degradation for HF and HML specimens but not for Teflon (PVC, of course, could not be evaluated in liquid helium). Again it is tempting to theorize that hydrolytic molecular scission is indicated by the decreased flexibility in liquid helium. However, physically absorbed moisture, rather than molecular scission, may be responsible for the decreased flexibility of the insulations in liquid helium.

Compression, Cut-through Resistance

Test methods for the cut-through characteristics of wire insulation have been the subject of more controversy than perhaps any other wire evaluation technique. The controversy arises at least in part because so many factors are involved and the mechanism of failure obtained is different not only for different materials but also with different test methods. It is not within the scope of work under the subject contract to solve the thermal cut-through evaluation problem. It is

Table XII

Repeated Mandrel Flexibility Test at 4°K

Test Conditions: 10 Reverse Bends Around Mandrel Diameter Shown While Under Liquid Helium, Failure Indicated By Low Power Microscope Visual Examination for Breaks, Cracks, Splits or Separation of Dielectric From Wire

		Pre-Condition Aging of Wires Before Repeated Mandrel Flexibility Test in Liquid Helium								
Insulated Wire		None (As Received)	120 C/Vac 20 Days	120 C/Vac 60 Days	120 C/Vac 120 Days	120 C/Air 60 Days	120 C/Air 120 Days	250 C/Air 60 Days	250 C/Air 120 Days	80 C/95% R.H. 15 Days
Teflon (Extruded) (0.0114" Wall)	Failure	1"	1"	1-1/4"	3/4"	1-1/4"	3/4"	1"	3/4"	1"
	Satisfactory	1-1/4"	1-1/4"	1-1/2"	1"	1-1/2"	1"	1-1/4"	1"	1-1/4"
Poly Vinyl Chloride (0.0071" Wall)	Failure	1-3/4"	1-3/4"	1-3/4"	1-3/4"	1-3/4"	1-3/4"	(No Test) (Above plastic flow temperature)	(No Test)	1-3/4"
	Satisfactory	--	--	--	--	--	--			--
Heavy Formex (0.0013" Wall)	Failure	1/2"	(No Test)	1/2"	1/2"	1-3/4"	1-3/4"	(Insulation Charred and Flaked Off at 250 C)		1-3/4"
	Crazed	1/2" - 1-3/4"	1"	1/2" - 1-1/2"	3/4" - 1"	--	--			--
	Satisfactory	1-1/4"	1-1/4"	1-3/4"	1-1/4"	--	--			--
HML (G.E. Co.) (0.0014" Wall)	Failure	None	None	None	None	None	None	1/2"	1/2"	1/4"
	Welts	--	--	--	--	--	--	1/8" - 3/4"	1/8" - 3/4"	--
	Satisfactory	1/8"	1/8"	1/8"	1/8"	1/8"	1/8"	1"	1"	1/2"
HML (Phelps-Dodge) (0.0011" Wall)	Failure	None	None	None	None	None	None	1/2"	1"	1/2"
	Welts	--	--	--	--	--	--	1/8" - 3/4"	1/8" - 1"	--
	Satisfactory	1/8"	1/8"	1/8"	1/8"	1/8"	1/8"	1"	1-1/4"	3/4"
Asbestos (Phosphate) (0.0054" Wall)	Failure	1/2"	3/4"	1/4"	3/4"	1/4"	3/4"	1/2"	3/4"	3/4"
	Cracks	1/8" - 1/2"	1/4" - 1/2"	1/2"	1/2" - 3/4"	--	3/4"	1/2"	1/8" - 3/4"	1"
	Satisfactory	3/4"	1"	3/4"	1"	3/4"	1"	3/4"	1"	--
Asbestos (ML Overcoat) (0.0064" Wall)	Failure	1/4"	1/2"	1/2"	1/2"	1/4"	1/2"	1/4"	1/2"	3/4"
	Cracks	1/8" - 1/4"	1/8" - 1/2"	1/8" - 1/2"	1/4" - 1/2"	1/8" - 1/2"	1/4" - 1/2"	1/8" - 1/4"	1/8" - 1/2"	1/4" - 3/4"
	Satisfactory	1/2"	3/4"	1/2"	3/4"	3/4"	3/4"	3/4"	3/4"	1"

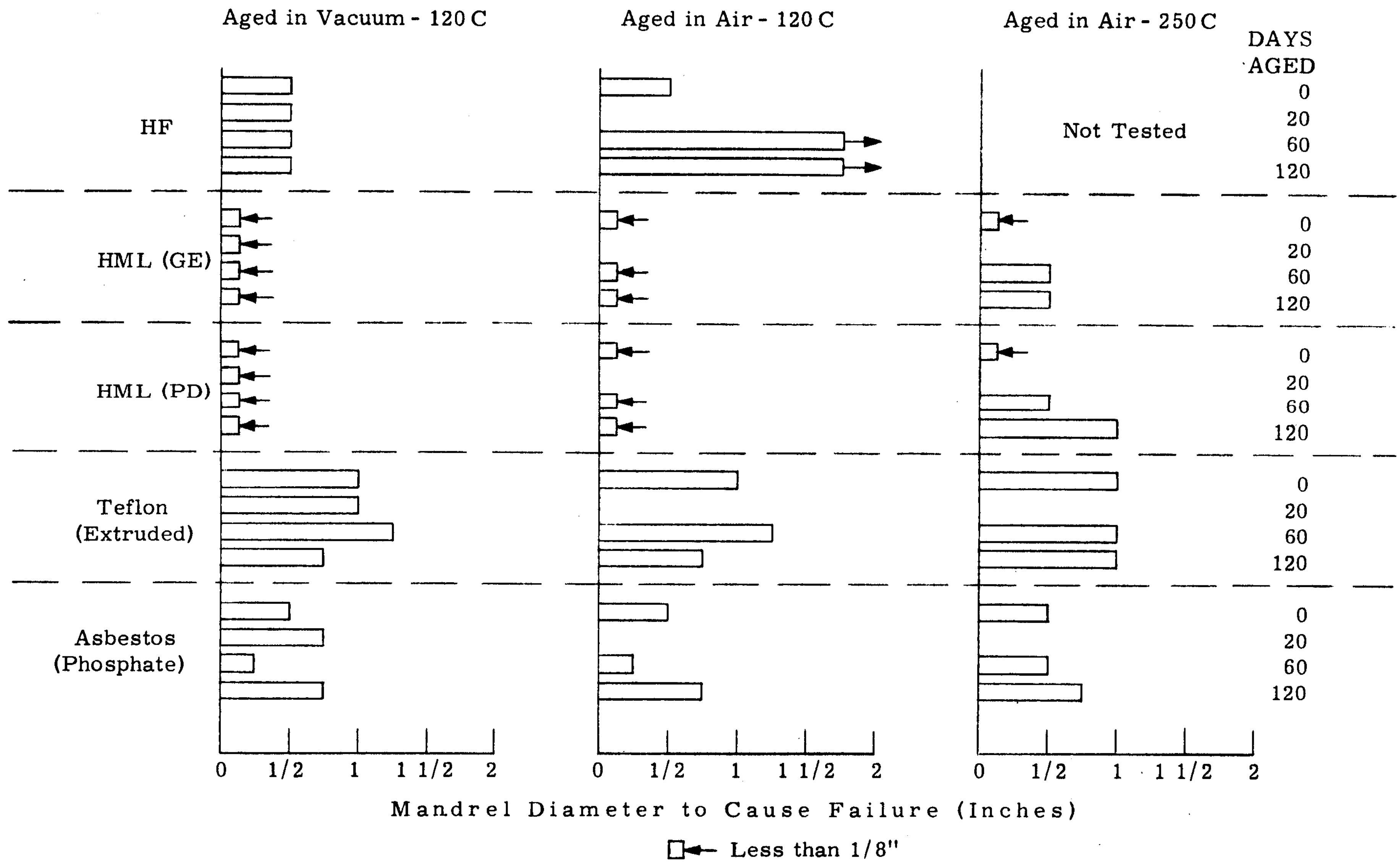


Fig. 9 Comparison - Repeated Mandrel Flexibility Tests at -269 C after Thermal Aging

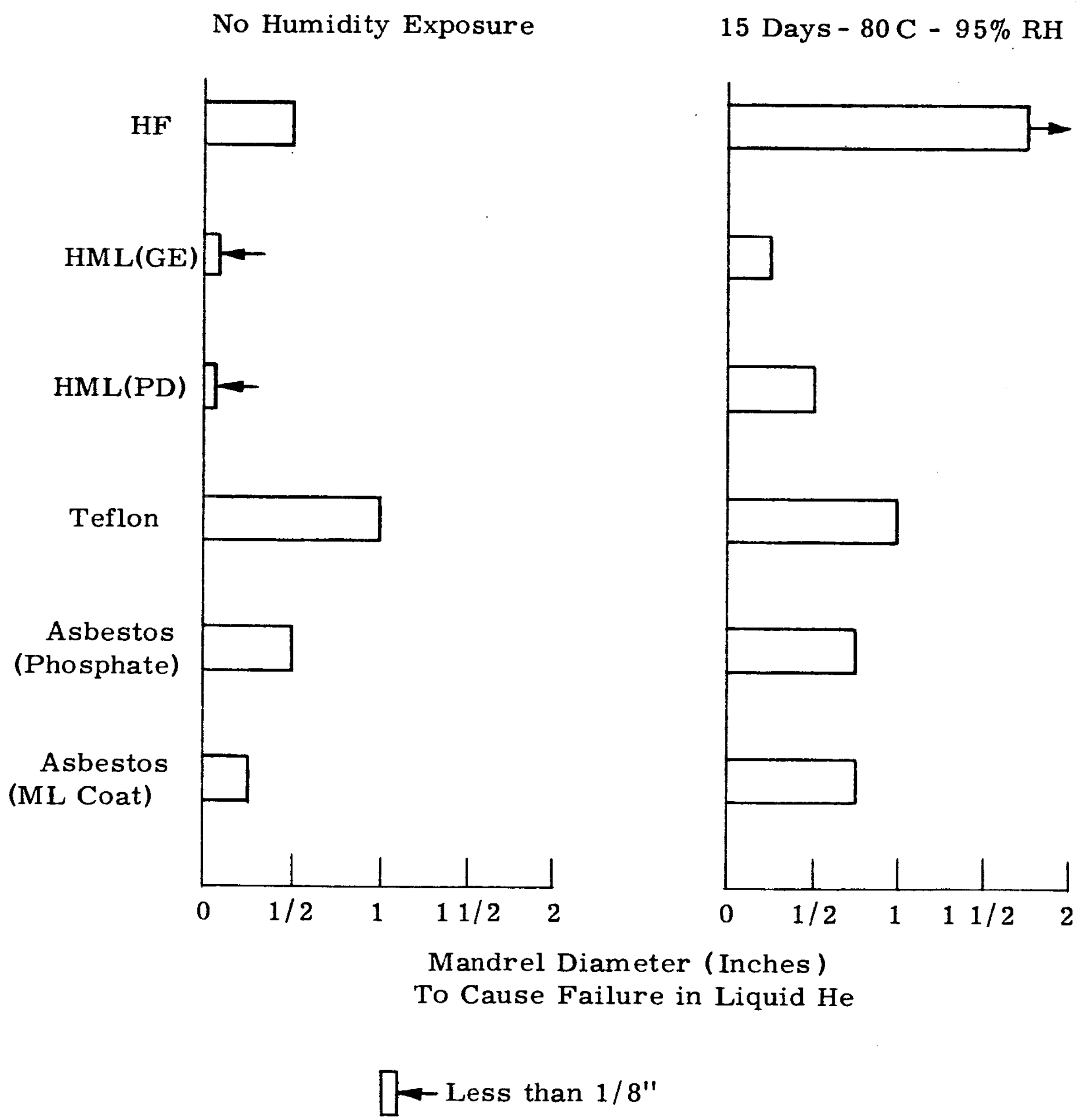
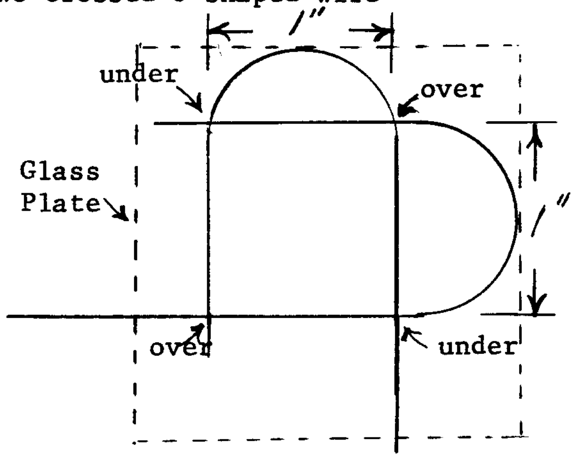


Fig. 10 Effect of Humidity Exposure on Repeated Mandrel Flexibility at −269 C

important, nevertheless, to gain some idea of the cut-through performance of insulations considered for cryogenic applications because in processing or in part of their life cycles they may be exposed to high temperatures. It is even more important to determine if failure under impression, stress is likely to occur at cryogenic temperatures as has been occasionally reported.

High temperature cut-through performance will be considered first. MIL 583 contains methods for thermal cut-through which involve crossed wires and a slowly increasing temperature. Using this method, HF fails at about 180C and HML does not fail at 500C (well beyond the limits of the test). The MIL test is not easily applied to extruded coatings like Teflon or PVC for which time as well as load and temperature is important. In this program, such "creep" effects have been qualitatively investigated as follows. Two crossed U shaped wire specimens are made as shown at the right and placed between 1/4 inch thick glass plates. Several samples may be stacked one over the other. The crossed wire specimens may then be placed in an oven, immersed in liquid nitrogen or kept at room temperature. A desired load may then be applied to the top of the stock and left for a specified time. The voltage breakdown between the two wire samples can then be measured. For a comprehensive evaluation the effect of temperature, load and time can be investigated. For this program the test was limited to 10 kilograms applied for 48 hours at 250, 120, 23 and -196C (in liquid nitrogen). Test results are given in Table XIII. The test parameters were chosen to be very severe so as to provide a sharp separation between the various insulations. The test results indicate that this objective was accomplished. For example, PVC cuts through at room temperature but not at -196C and Teflon cuts through at 120C but not at room temperature. HF fails at 250C but not at 120C and HML does not fail at all. Examination of the samples indicates that true thermoplastic flow occurs with Teflon and PVC (the PVC sticks together at 120C but not at room temperature). However, Formex at 250C appears to fail by rupture rather than true flow of the enamel film (of course after 48 hours at 250C considerable thermal degradation has occurred in Formex, also). All of the samples showed considerable deformation of the copper but less at -196C. (The asbestos insulated specimens also did not exhibit as much copper deformation as the other materials.)



It is apparent from the foregoing that plastic flow in materials like PVC and Teflon is greatly reduced at liquid nitrogen temperatures or below. Instead, the brittleness might lead to a "shattering" failure under compressive stress at low temperatures. Such failure was obtained in liquid nitrogen with a 4.7 lb. load applied to a .025 in. diameter steel rod held perpendicular to a PVC insulated wire. Cracking and spalling occurred also in a similar test on HF in liquid nitrogen while

Table XIII
Breakdown Voltage - KV
Crossed Loop Compression Tests

(48 hours under 10 Kg load)

Wire	Wall Thick- ness - In.	-196C (Liq. N_2)	23C	120C	250C
HF	.0013	Not Tested	Not Tested	8.1 8.5 11.0 9.2	Shorted
HML(GE)	.0014	8.5 9.3 9.3 9.0	Not Tested	12.3 14.2 15.6 10.7	10.5 12.0 12.0 11.5
PVC	.0071	15.8 *	0.1 0.2 0.15	Shorted	Not Tested
Teflon	.0114	16.8 27.0 31.0 25.0	14.1 15.0 16.0 15.0	Shorted	Shorted
Asbestos (Phosphate)	.0054	Not Tested	Not Tested	1.0 1.4 1.4 1.3	0.9 1.0 1.2 1.0

* Two other tests were abortive because the glass separating plates broke.

a load was being applied slowly to a maximum of 50 pounds. Unfortunately, the load at which failure actually occurred could not be determined by other mechanical or electrical techniques. The voltage breakdown, for example, was not markedly reduced when cracking occurred probably because liquid nitrogen - a good dielectric - instantly filled the cracks. Many techniques have been investigated, without success, in an attempt to obtain a quantitative measure of shatter resistance as described in the 7th Quarterly Report. The problem is complicated further by the necessity for careful microscopic examination to determine the presence of cracks when they do occur in some materials like HML. For example, a 500-pound load applied to a hardened .025 in. steel rod held at right angles to the HML insulated wire in liquid nitrogen will nearly cut the wire in two as shown in Photo #1. Examination with a low power microscope will fail to show any damage to the ML enamel film. However, electrical tests of the test specimen in salt water will indicate that film failure has occurred. With careful metallurgical techniques the sample can be cast in plastic, cross-sectioned, polished, etched, and photographed under high-magnification as shown in Photo #2. It is then possible to see the cracks in the enamel film and evidence of loss of adhesion. The same technique has been used to show the absence of cracks in an HF film also placed in compression under liquid nitrogen but with much less compressive load as shown in Photo #3. If the compressive deformation is made severe enough, HML and other wire enamels can be made to crack even at room temperature as shown in Photo #4. In this case, 50 pounds was applied to a .010 in. diameter needle held at right angles to the wire. Careful examination of the photograph indicates, also, that the enamel film under the needle has delaminated and the outer layer or layers have flaked away. The lamilar structure of the wire enamels is clearly visible in several of the microphotographs. It can be noted here that delamination has been noted occasionally with some HML films on wire in mandrel flexibility tests under liquid helium.

The microphotographs indicate that with laborious experimental techniques, the tendency for wire insulation to crush under compressive load at very cold temperatures can be determined. It is evident, also, except for PVC, very large compressive stresses which cause considerable deformation of the copper are needed to cause degradation. It is doubted that these severe stresses are representative of usual application conditions but do provide warning that they should be avoided.

Abrasion Resistance

If cut-through tests are subject to the greatest disagreement, abrasion tests for insulated wires are undoubtedly next in line. Again, it is not the purpose of the subject work to develop abrasion tests but rather to obtain at least a qualitative idea of performance in this respect. Other work has indicated that the NEMA repeated scrape abrasion test can be used with extruded insulations as well as the film coated wire for which it was designed. It is necessary to change the loads on the machine to cover a range of wire insulations. Consequently, as given in Table XIV, the standard NEMA loads have been

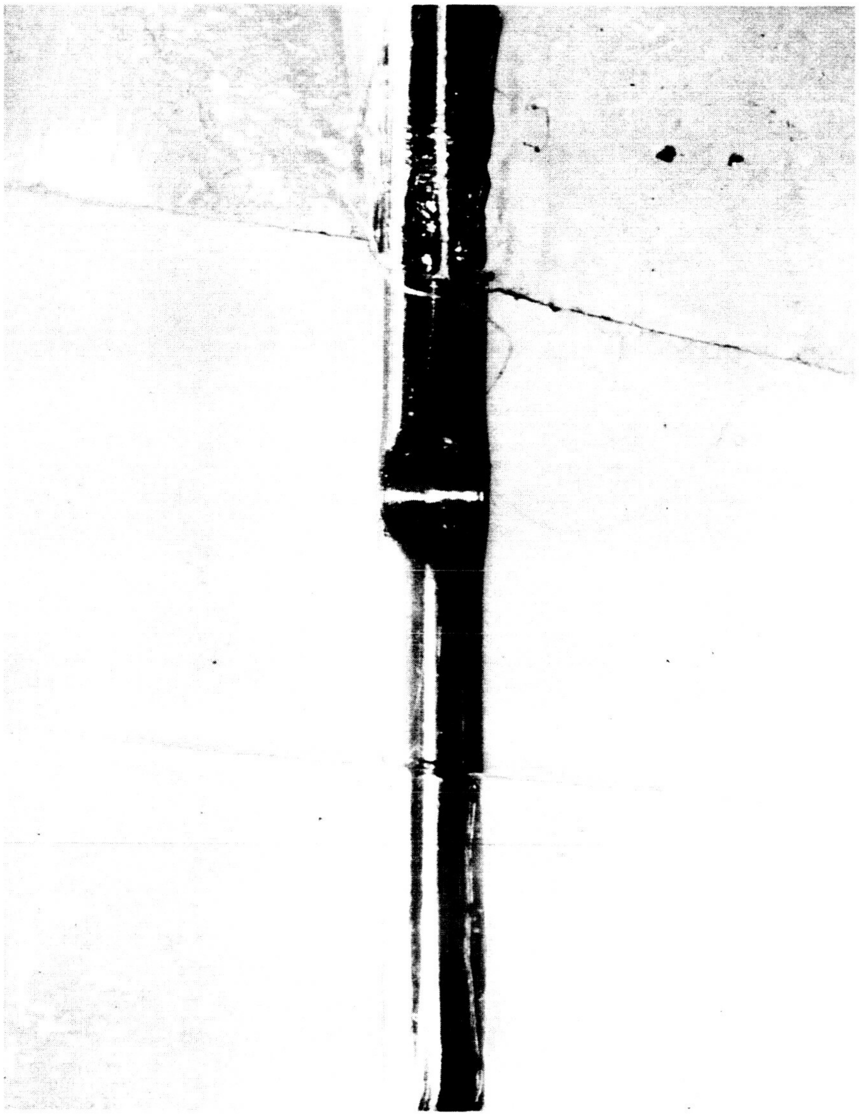


Photo #1

Depression in .025 in HML Insulated Wire Produced by 500 Pound Load on .025 in Steel Mandrel in Liquid Nitrogen - 20X Magnification

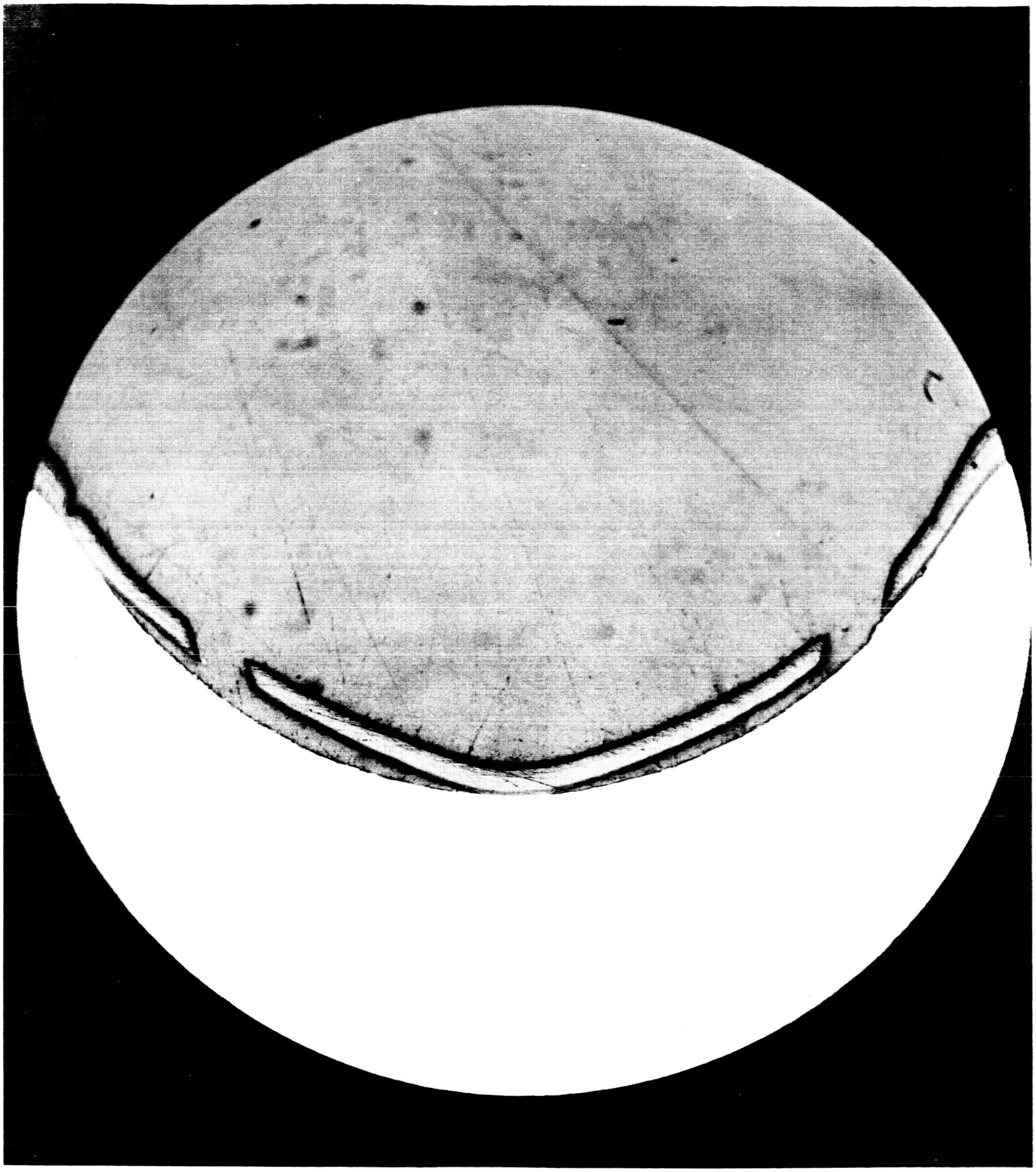


Photo #2

Cross Section of Depression in .025 in HML Insulated Wire Produced by 500 Pound Load on .025 in Steel Mandrel in Liquid Nitrogen - 250X Magnification

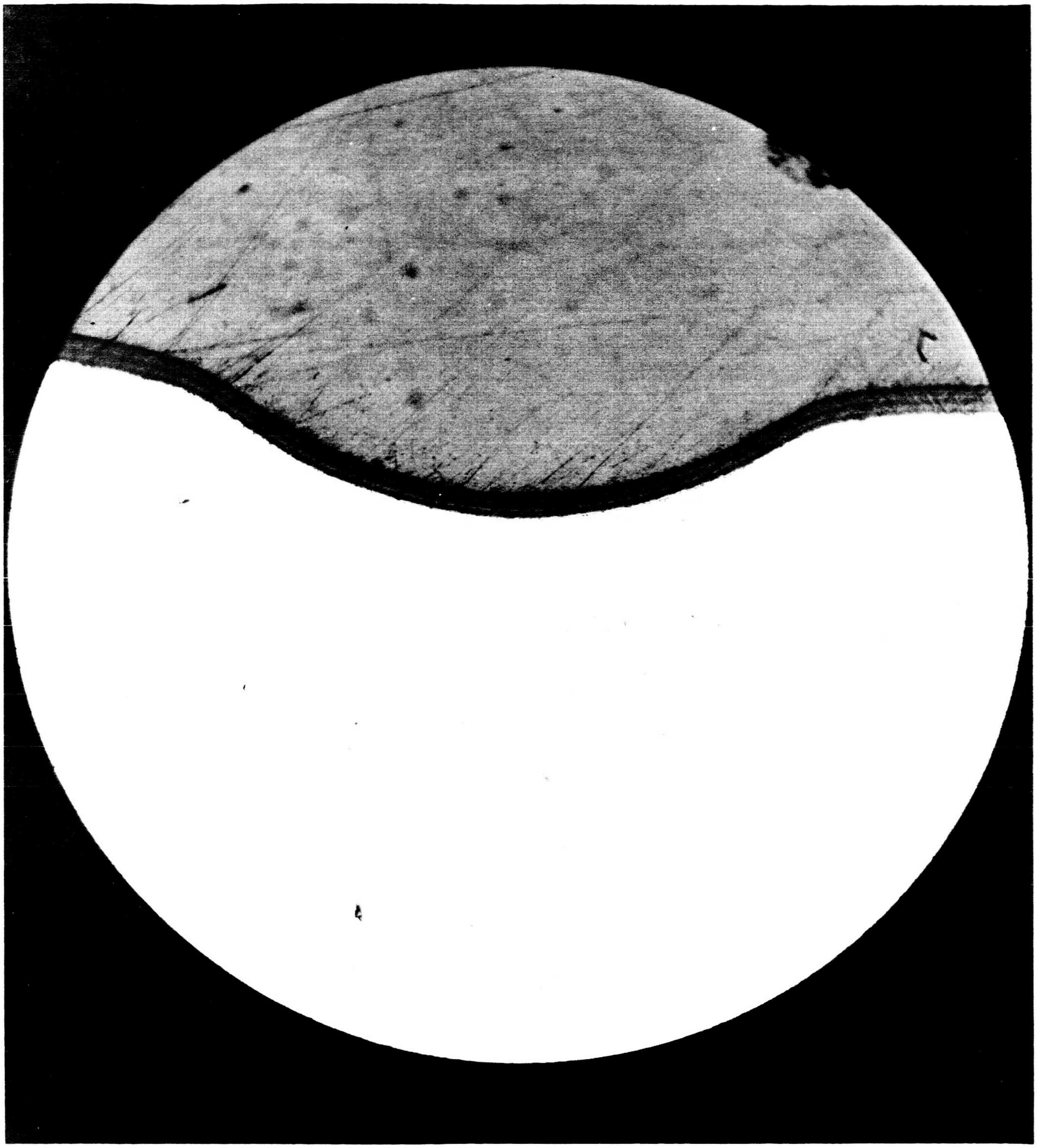


Photo #3

Cross Section of Depression in .025 in HF Insulated Wire Produced by
10 Pound Load on .025 in Steel Mandrel in Liquid Nitrogen - 250X Magnification

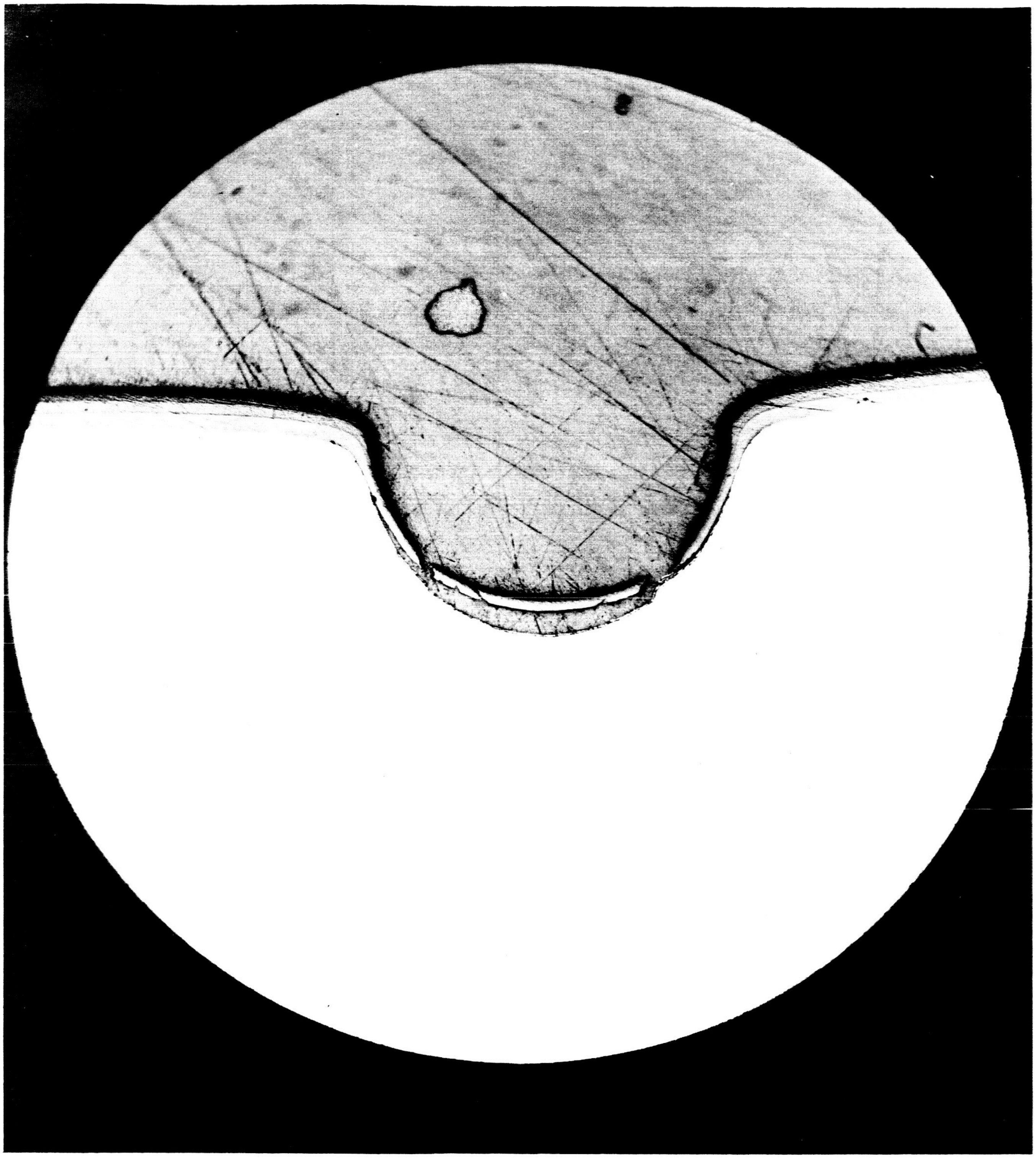


Photo #4

Cross Section of Depression in .025 in HML Insulated Wire Produced by 50 Pound Load on .010 in Needle at Room Temperature - 250X Magnification

Table XIV

Repeated Scrape Abrasion Tests at 23C 50% RH

Number of Cycles to Failure

	Load - 540 grams					
	HF	HML(P.D.)	HML(G.E.)	PVC	Teflon	Asbestos
	100	25	18	16	> 500	3
	62	35	24	11	> 500	4
	87	20	85	10	> 500	2
	27	31	70	14		4
	252	33	48	12		2
	90					
	106					
Avg.	103	29	49	13	> 500	3

	Load - 270 grams	
	PVC	Asbestos
	28	3
	17	6
	24	8
	24	5
	23	5
Avg.	23	5

	Load - 1000 grams
	Teflon
	75
	102
	30
	48
	73
Avg.	66

used with the film coated wires HF and HML, a higher load also with the extruded Teflon and a lower load also with PVC and aluminum phosphate impregnated asbestos. The variability in the test results is normal. Despite the somewhat lower repeated scrape abrasion of HML as compared to HF, such a difference is not usually apparent in the service experience known to the author. The superiority shown for the Teflon will undoubtedly be realized in application. The deficiency in the abrasion resistance of the aluminum phosphate impregnated asbestos is also considered realistic in terms of service.

Mandrel Flexibility of Ribbon Cable

No further work on ribbon cable has been carried out during the last quarter because the desired samples of H film construction have not yet been obtained. The results described in the Seventh Quarterly Report, therefore, have been included here in Table XV. It is apparent that superior performance is contributed by H film and that the attempts to obtain superior H film constructions should be continued.

Dielectric Constant and Dissipation Factor of Cryogenic Liquids

The interesting and in some respect surprising studies on the breakdown voltage of cryogenic liquids (see 4th Quarterly Report - July 16, 1962) have indicated a need for an understanding of other electrical properties. A search of the literature has revealed data on the static or low frequency, dielectric constant of cryogenic liquids but no information on their dielectric losses. A conversation with Prof. Van Itterbeek in Louvain, Belgium, indicates that at least some of these dielectric constant determinations were made by calculation from DC absorption measurements. Such calculations would give the so-called "static" dielectric constant. A zero frequency dissipation factor can be calculated also but for very low values it is doubted that sufficient sensitivity could have been obtained in instruments available at the time the measurements were made.

While the "static" or zero frequency measurements are interesting, they are most useful in considering the ultimate extension of the low frequency range. For this reason, measurements were attempted of dielectric constant and dissipation factor in the range of frequencies from 100 to 10,000 cps. When measurements must be made at the bottom of a cryostat, the use of a guard circuit to eliminate lead leakage and other errors is a practical necessity. Except for a few Schering bridges operating primarily at 50 or 60 cps, guarded AC bridges with great sensitivity have not been available until very recently. Fortunately, the very new General Radio Type 1620-A Transformer-Radio Arm Bridge does meet these exacting needs.

Table XV

Repeated Mandrel Flexibility Tests of Ribbon Cable

Test Condition: Strips 3/16" Wide Were Carefully Cut from the Cable for Mandrel Winding in a Manner Similar to the Procedure Developed for Round Wire. 10 Reverse Turns in Liquid Helium was the Criterion for Failure

Sample No.	Material	Results	Mandrel Dia. - In.	Remarks
A	Methode Plyoduct (PD-812-P4) (31/32" Wide x 0.012" Thick, 12 Copper Conductors) (Mylar)	Failed Satisfactory	1/2" 1"	(If stress relieved by pre-flexing at Room Temperature)
B	Polystrip (H-100-C-25) (2-5/8" Wide x .0086" Thick, 25 Conductors) (Resin Bonded H Film)	Failed Satisfactory	-- 1/4"	(Could not be wound on 1/8" Mandrel) (Except at point of high stress near mandrel attachment)
C	Polystrip (P-100-C-12) (1-5/16" Wide x .0085" Thick, 12 Conductors) (Resin Bonded Mylar)	Failed Satisfactory	1/4" 1/2"	 (Except at point of high stress)
D	Polystrip (TX-156-C-20) (3¼" Wide x .012" Thick, 20 Conductors) (FEP Teflon)	Failed Satisfactory	1" --	(Test strip 1-1/16" wide)
E	IRC-HX-100-C-12 (1-5/16" Wide x .012" Thick, 12 Conductors) (.002 H Film/ .002 FEP Teflon - .005 FEP Teflon - heat bonded)	Failed Satisfactory	-- 1/4"	(Could not be wound on 1/8" Mandrel)

A guarded liquid cell capable of evacuation to a high vacuum is also needed for the evaluation of cryogenic liquids. Mr. J.M. Atkins has solved this problem ingeniously by the adaptation of a standard 1000 picafarad vacuum capacitor as shown in Fig. 11. The capacitor was obtained from the manufacturer with the long glass evacuation tube in place. The tube was sealed off several inches above the capacitor and then reconnected in the vacuum system associated with the cryostat. The completely guarded, cryogenic liquid cell is shown in Photo #5.

Test results are given in Table XVI and the dielectric constant value from the literature is included for comparison. It is interesting that close agreement with the literature has been obtained for hydrogen and helium but that the values for nitrogen differ. It is of course possible, but is not considered likely, that the nitrogen in these tests may have been contaminated with oxygen. Great efforts were made to avoid contamination and the test cell was evacuated to a 10^{-6} torr. The values of dissipation factor (tan δ) are so low that they must be considered more in the nature of indications rather than accurate values. (The two values shown were obtained with the bridge connected in two different ways.) At the lower frequencies the values were very difficult to obtain. However, trends are apparent with frequencies which are interesting. The dissipation factor of both liquid nitrogen and liquid hydrogen increases with an increase in frequency. The dissipation factor of helium, on the other hand, decreases with an increase in frequency. Helium contaminated with a small amount of air, however, has a characteristic like that of nitrogen rather than that of helium alone.

Within the limits of recognized, questionable accuracy, the values obtained indicate interesting differences in the loss characteristics of cryogenic liquids. It will be particularly interesting to measure the dielectric losses of super fluid helium and such measurements are planned for the future.

PROGRAM FOR JULY AND THE NINTH QUARTER

The preparation of this report and the full report on the European visits will constitute the principal activity for the month of July.

In the remainder of the ninth quarter, the development of bundled conductors and improved ribbon conductors for cryogenic applications will be initiated. The program for this development will be detailed in the monthly report for July. Voltage breakdown measurements on very pure cryogenic liquids will be undertaken also during the coming quarter. The special test fixtures for this work are now available. The need for the work has been emphasized by information obtained during the European visits described in the appendix to this report.

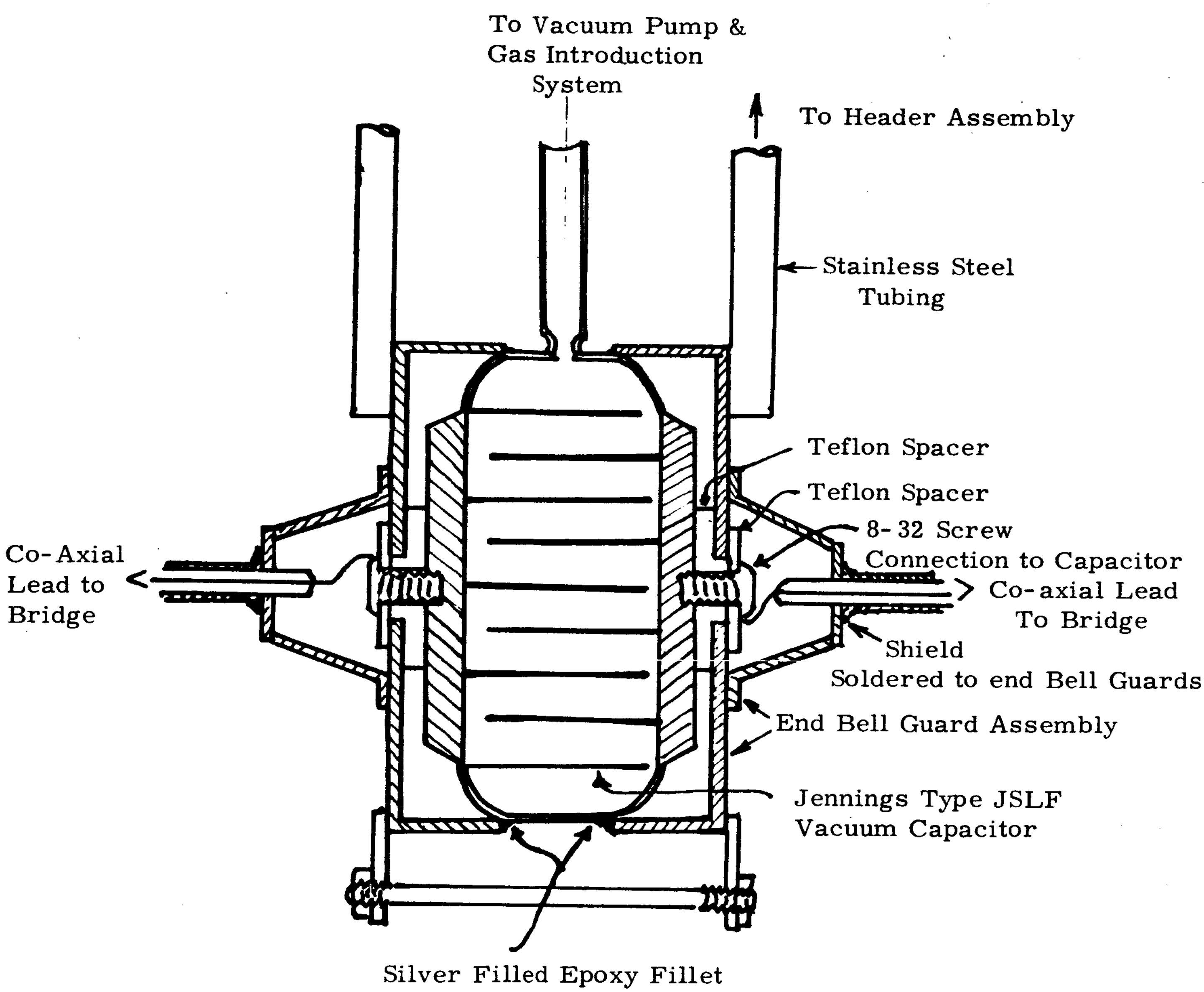


Fig. 11 Sketch of Dielectric Cell for Cryogenic Liquids

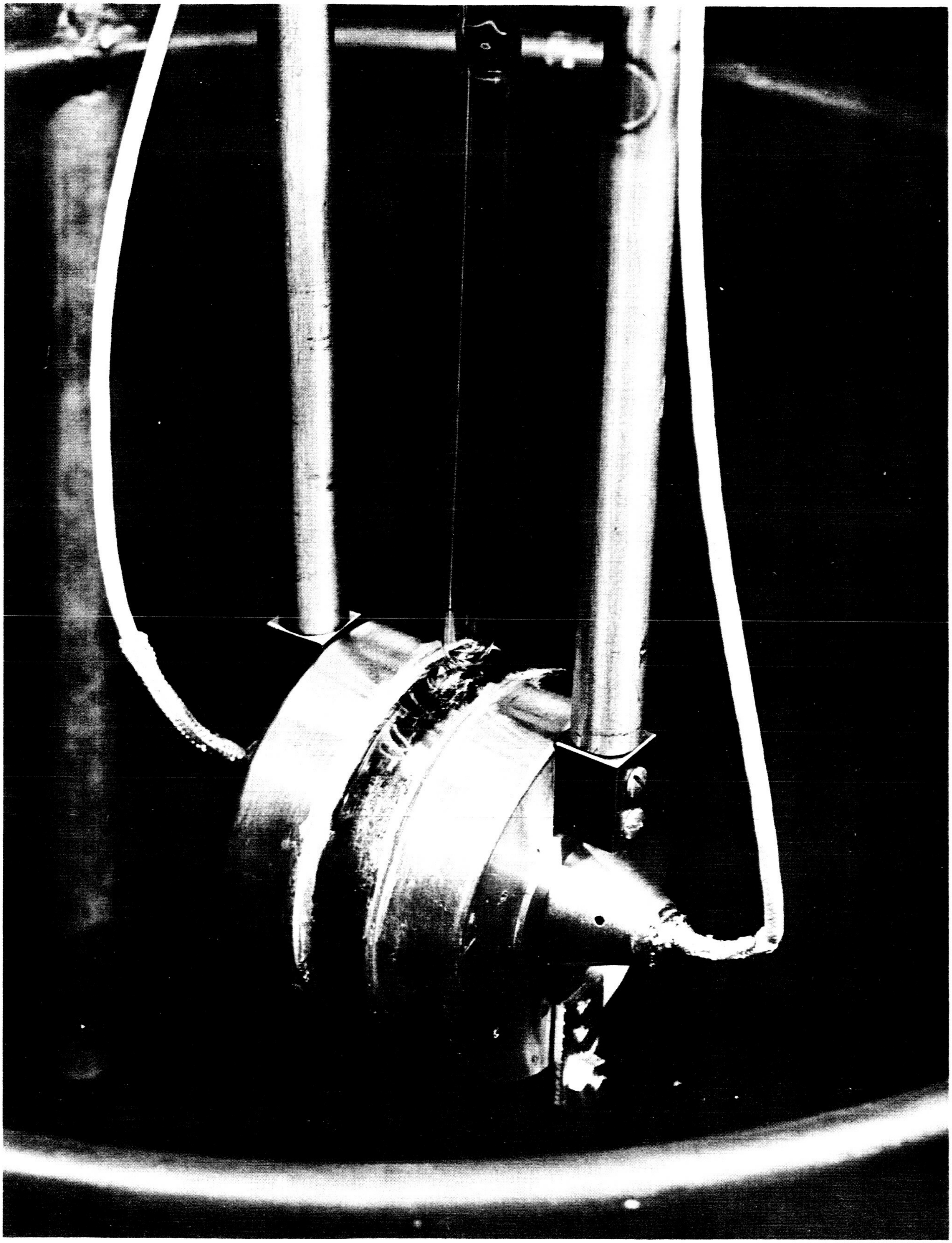


Photo #5

Completely Guarded and Evacuated Cell for Measurement of Dielectric Constant and Dissipation Factor of Cryogenic Liquids

Table XVI

Dielectric Constant and Dissipation Factor

of

Cryogenic Liquids

Liquid	Pressure mm.	^{o}K	ϵ' Meas.	ϵ' Lit.*	Meas. $\tan \delta$	f cps.
Nitrogen	746.8	77.3	1.462	1.433^{8}		100
			1.462			500
			1.462^{3}		.0000058, .000016	1,000
			1.462^{3}		.000010, .000015	5,000
			1.462^{2}		.000020, .000031	10,000
Hydrogen	746.8	20.3	1.225	1.226		100
			1.225		.000010 (?)	500
			1.225			1,000
			1.225		.000005, .000010	5,000
			1.225		.00001 .000036	10,000
Helium	745.2	4.2	1.048	1.048^{0}		100
			1.048		.000020	500
			1.048		.000012	1,000
			1.048^{6}		.0000034, .0000050	5,000
			1.048^{6}		.000000 .000007	10,000
Helium Contaminated with air	751.1	4.2	1.051^{8}		.00000 (?)	100
			1.051^{8}		.0000034	500
			1.051^{8}		.0000018	1,000
			1.051^{8}		.000010, .000013	5,000
			1.051^{8}		.000020, .000028	10,000
Oxygen		77		1.515		

* Value from the literature - Frequency of measurement not stated - see references attached.

References

R.B. Scott, "Cryogenic Engineering", D. Van Nostrand, Princeton, 1959.
N_2 -- Robert Guillien, J. physique (8), 1, 29 (1940).
H_2 -- A. Van Itterbeek & J. Spaeden, Physica (9), 339, (1942).
H_e -- W.M. Keesom, "Helium", Elsevier, Princeton, 1942.
National Bureau of Standards, Circ. 514.

APPENDIX A

European Visits
to
Cryogenic Laboratories

INTRODUCTION

As a part of a European trip, the author took the opportunity to visit a number of European Laboratories involved in cryogenic studies. So far as possible, these laboratories were selected on the basis of outstanding contributions to cryogenics in general and, in particular, to the study of cryogenic liquids themselves or the properties of nonmetallic materials at cryogenic temperatures. It was recognized in advance that no other work exactly duplicating the subject contract might be found. The visits were undertaken in order to determine what information - published and unpublished - was in fact available with a direct or indirect relationship to the subject work. As a secondary objective, the visits were designed to stimulate interest in the properties of insulating materials and plastics at cryogenic temperatures and to encourage the exchange of such information in the future. During each visit a special effort was made to obtain information about similar work by others.

The following summary of the European visits has been appended to this annual summary report because it is a part of the last year's activities and the amended contract requirements. Time has not been available, by the dead-line for this report, to prepare a detailed report of the European visits including referenced information. The detailed report will be made just as soon as possible.

SUMMARY AND CONCLUSIONS

The traditional centers for cryogenic studies, such as Cambridge, Oxford and perhaps Leyden, are apparently emphasizing theoretical studies of solid and liquid structure and also super conductivity. Important as this work is, it is of only incidental interest in reference to nonmetallic materials. Theoretical studies of cryogenic liquids are well advanced in a number of European laboratories and these studies are of at least indirect interest for the background on cryogenic fluid technology which they provide. The excellent voltage breakdown and ion mobility studies in cryogenic liquids now underway in several European laboratories are scientific rather than engineering in nature but provide technical background of direct importance to the GE-NASA engineering program.

The beginnings of an interest in cryogenic properties of nonmetallic materials is apparent in industry or in the industry-supported plastics institutes rather than in the European universities. The work on the cryogenic properties of nonmetallic materials has also been undertaken primarily with scientific purposes in mind. Again, a technological background for sound engineering work is thereby developed. However, the stress-strain characteristics of plastics down to nitrogen temperature, which are being developed in at least two laboratories, are primarily of engineering interest. The need for continuing exchange of information is established.

No specific program for the development of wire and cable insulation in cryogenic applications appears anywhere in the European picture. Even indirect observations on the performance of such insulations are extremely limited. If the lack of such programs is disappointing, the need for the GE-NASA program is the more obvious. There is evidence that not only scientific, but engineering studies also, on the properties of insulating materials and plastics at cryogenic temperatures will multiply and expand in Europe. The development of industrial cryogenic devices will accelerate such a trend. The motivation for cryogenic studies with nonmetallic materials at present comes to a considerable extent from interest in liquid fuel systems associated with space programs. Only the United States, the USSR, France and perhaps Great Britain, so far, have a major stake in space.

SUMMARY OF VISITS

The following summary gives the name of the cryogenic laboratory visited, the principal individuals contacted, principal areas of work and pertinent observations relating to work on nonmetallic materials or cryogenic liquids.

Central Electricity Board Research Laboratories
Leatherhead, England -- May 13, 1963

Dr. P.F. Chester
Dr. R.A. Kamper

The primary work concerns study of "hard" superconductors and high field, superconducting magnets. Melinex (a polyester film like Mylar) has been used successfully in liquid helium but Westinghouse (USA) problems with voltage breakdown of Mylar were mentioned. No studies specifically directed to cryogenic performance of insulating materials or plastics have been undertaken or are planned.

The high field cryogenic magnets are of interest in magnetohydrodynamic studies. A "paper" study of cryogenic transformers by AEI - Rugby was mentioned. Chester did not believe that any industrial laboratories in Britain were investigating cryogenic properties of nonmetallic materials but recognized a need for such studies. (Note: The author confirmed this statement during later discussions with R. Snadow of AEI, Rugby.)

Electrical Research Association Laboratories
Leatherhead, England - May 13, 1963

Mr. Waters - Deputy Head, Transformer Dept.

No experimental cryogenic studies of any magnitude are in progress. A "paper" study of cryogenic transformers has indicated lack of design feasibility. Similar studies by AEI and Parsons in England were reported.

National Physical Laboratories
Teddington, England - May 14, 1963

Dr. W. Davies	Dr. C. Stoddard
Dr. Stuart	Dr. Catteral
Dr. J.S. Hill	Dr. R.W. Powell

The principal effort in cryogenics at NPL is concerned with cryogenic switching (Cryotrons) under Dr. Stuart's direction and the thermal conductivity studies by Dr. Powell. In the cryotron work trouble with electrical leads at cryogenic temperatures has lead to limited developmental work. Teflon has been found useful but still subject to brittle failure. Extruded Nylon was found to be much less serviceable than Teflon. Troubles have been encountered at cryogenic temperatures with gaskets also -- particularly with silicone rubber.

Dr. Stoddard and others are studying vapor deposited films of SiO which have been shown to shift the critical temperature of tin films. In consequence, the in-situ polymerization of resin films for insulation at cryogenic temperatures are underway. Cryogenic adhesion problems of the organic films to the substrate and the overlying thin metal films has received considerable study without adequate solution.

R.W. Powell has developed suitable methods and carried out thermal conductivity measurements on nonmetallic materials at cryogenic temperatures. He has followed the work of others in his field and collected exhaustive references. (A considerable list of references provided will be included in the full report.) A three ball "thermal comparator" has been developed for making rapid thermal conductivity tests over a very wide range of temperatures.

University of Cambridge, Dept. of Physics, Magnetic Lab.
Cambridge, England - May 15, 1963

R.L. Powell (Note: D. Schoenberg was unavailable but the author had a brief conversation with him on May 17 at Oxford.)

R.L. Powell, on leave from the National Bureau of Standards in Boulder, Colorado, acted as guide in describing the work under Dr. Schoenberg's general direction. The entire program at Cambridge seems to be directed to very fundamental cryogenic studies of metallic structure --

both normal and superconducting ultrasonic absorption, microwave (2 mm) absorption, electron spin resonance (ESR) as well as other techniques are being used. Schoenberg's greatest personal interest is now directed to "Fermi surface" studies using a pulsed 200 Kilogauss magnetic field. Westinghouse (USA) has given Cambridge a cryomagnet for such studies, also.

Cambridge has apparently done no work on nonmetallic materials and does not seem to be interested in it. No information about the work others in the nonmetallic area could be obtained.

<u>Queen Mary College (University of London)</u>
London, England - May 16, 1963

Dr. T.J. Lewis - Dept. of Electrical Engineering
Dr. B. Salvage - Dept. of Electrical Engineering
Prof. Humphrey Davies - Head Dept. of Electrical Engineering
Dr. D.H. Martin - Dept. of Physics

Dr. Lewis heads the extremely competent and extensive work on the electric breakdown of liquids including the cryogenic liquids, nitrogen, oxygen, argon and methane. So far no work has been done with liquid helium but reference was made to the work of Edwards in the USA and particularly to that of Prof. Blaisse in Delft (see later). Lewis has been particularly concerned with the influence of impurities and the condition of electrodes on breakdown voltage - details will be given in the full report. The character of the electrodes may change the intrinsic electric breakdown voltage of cryogenic liquids by as much as 4 to 1.

Investigations of ion and electron mobility in liquid argen and methane have been productive. (The work of Careri in Rome was mentioned - see later.) The attachment coefficients of oxygen in liquid argon and methane have been studied.

Great interest was evidenced in the G.E. work on Cryogenic liquids -- particular in the dissipation factor measurements. Dr. Lewis and others believe that the whole area of nonmetallics at cryogenic temperatures needs extensive investigation and appeared eager to continue exchange of information. The facilities at Queen Mary College are modern, excellent and amazingly extensive. Sponsored work in the area should be competent and efficient.

Dr. Martin is specializing in far infrared absorption in liquid helium and solid argon. Both dielectric and mechanical absorption studies are being made, also, particularly with solid argon.

<u>Oxford University, Clarendon Laboratories</u>
Oxford, England - May 16, 1963

Dr. H.M. Rosenberg (Dr. K.Mendelssohn was away in London.)

Dr. Rosenberg served as a guide to introduce seven graduate students who described their programs. Dr. Rosenberg and Mr. Lewis are interested in the low voltage breakdown strength of liquid helium because of electrical failures they have experienced in ultrasonic microwave studies. The work in Dr. Mendelssohn's area, like the work at Oxford, is highly theoretical and directed primarily to studies of superconductivity. Some of the work, however, is directed to the study of physical properties of the cryogenic liquids and to design of very low temperature (0.4 K) or precise, temperature controlled cryostats. No work is in progress on nonmetallic materials at cryogenic temperature and little interest was expressed in it.

<u>Physikcalisch-Technische Bundesanstalt (PTB)</u>
Braunschweig, Germany - May 20, 1963

Dr. M. Kerston, President PTB	Dr. Kramer
Dr. E. Richter	Dr. Ruehl

Drs. Kramer and Ruehl are carrying on primarily an investigation of superconductivity in thin metal films. In particular, they have been concerned with the influence of absorbed gases on superconductivity. Liquid hydrogen, as well as liquid helium, is made and used at PTB. Interest was expressed in the dielectric properties of cryogenic liquids. Dielectric work by Prof. H. Pick, Technische Hochschule, Stultgart, on single crystals of anthracene in liquid helium was mentioned.

<u>Institut für Technisch Physike, Technische Hochschule</u>
Braunschweig, Germany - May 20, 1963

Dr. G. Schneider

Dr. Schneider regreted Prof. Justi's absence on business and described work on properties of semiconductors (metallic) at cryogenic temperatures. The conversation was somewhat difficult since it was carried out in rapid German (Dr. Schneider did not speak English.) Dr. Schneider stated that no insulation or adhesion problems have been encountered at cryogenic temperatures. Special rubbery, cellulose acetate adhesives are used very successfully. (UHU-Hart and UHU-plus made by M. Fischer, Bühl (Baden) Germany).

Little interest was expressed in nonmetallic materials at cryogenic temperatures.

<u>Deutsches Kunststoff-Institut</u>
Darmstadt, Germany - May 22, 1963

Prof. K.H. Hellwege, Director
Dr. W. Knappe
Dr. G. Langbein

Dr. Knappe described and showed the work of the Institute which covers a wide range of engineering and scientific studies on plastics. Thermal conductivity measurements are routinely made down to liquid nitrogen temperatures and occasionally, to liquid hydrogen temperature. Specific heat measurements over a wide temperature range are just being started. English work in this area by Danton and Evans Hall was mentioned. Electron spin resonance (ESR) techniques are being used to study free radical formation with Van de Graf radiation in both polyethylene and polypropylene down to liquid nitrogen temperatures.

Dr. Langbein is studying dielectric absorption in polymers down to -160C and mentioned work by Reddish of England, also. This work is to be quite theoretical with interest in very slow crystallization velocities, β transition temperatures, etc.

Great interest was expressed in the G-E work on properties of nonmetallic materials at cryogenic temperatures. The opportunity for continued exchange of information was evident.

Farbwerke Hoechst, A.G.
Frankfurt, Germany - May 22, 1963

Dr. Carlowitz

Hoechst has made stress-strain measurements of thermoplastics down to -100C and lower (data in full report). Elongation at break is 5% or less at low temperatures with interesting differences between materials of different molecular weights. Hoechst has made no dielectric measurements at very low temperatures but mentioned the work of Dr. Thurn at BASF in Ludwigshafen.

Despite the limited work at Hoechst, great interest was expressed in the low temperature properties of plastics and in the G-E work.

Institut für Tief Temperatur Forschung
Heersching (near Munich), Germany - May 24, 1963

Dr. R. Doll

The institute's activities are directed primarily to a study of hard superconductors and associated phenomena. A new expansion machine for production of liquid helium is under development. No work is underway on nonmetallics but interest was expressed in such studies. The work of Dr. W. Wiedemann at the Reaktorstation in Garching near Munich was mentioned. This work involves the effect of radiation at cryogenic temperatures on thermal and electrical resistivity as well as physical expansion on both metallic and nonmetallic materials.

Instituto di Fisica, University of Rome
Rome, Italy - June 10, 1963

Dr. G. Careri

Dr. Careri has a theoretical interest in the physics of liquids. He has studied ion mobility in liquid helium as a function of temperature and found mobility to increase as temperature is decreased below the λ point. Great interest was expressed in the G-E work on the dielectric characteristics of cryogenic liquids. Careri emphasized the importance of purity in the study of helium and suggested filtering superfluid helium through the finest Millipore filter (ordinary helium is too viscous!). He discussed other methods of purification for cryogenic liquids, also, which may prove to be of great importance in the G-E work.

Other cryogenic work at Rome includes nuclear magnetic resonance (NMR) studies of helium absorbed on zeolites, calorimetry to measure heat of absorption with helium III and other basic work on cryogenic liquids. Work on superconductivity is also in progress in another laboratory but was not discussed.

Research Laboratories of L'Air Liquide
Sassenage, France - June 17, 1963

Emile Carbonell
Alain P. de la Harpe

This new laboratory is concerned with basic research in cryogenics, development and prototype manufacture of storage and transport facilities for liquid hydrogen and oxygen and space research projects, including space simulation. The French interest in space projects was very evident.

Since the laboratory is so new, most of the research programs in the cryogenic area are just starting, including, for example, a study of electrical conductivity in super pure aluminum (impurity less than 25 ppm) with the object of achieving very high conductivities. Work on Peltier cooling is also starting. A small 30 Kgauss cryogenic magnet has been constructed. No work is planned on nonmetallics but considerable interest was shown. A very thorough study of cryogenic developments - the world over - has apparently been undertaken. Considerable information on the measurement of impurities was said to be available and sources were given. French work on super-conducting gyros was mentioned also.

Centre de Rechereches Sur Les Très Basses Temperatures
Grenoble, France - June 17, 1963

Prof. L. Weil

In a very brief meeting, Prof. Weil indicated relatively little interest in cryogenic studies of nonmetallic materials or in the electrical properties of cryogenic liquids. He did mention an unusual affinity of liquid helium for small amounts of moisture as an explanation for the dielectric losses in liquid helium. Some study has been made, also, on improved thermal transfer in cryogenic liquids with electrical fields of high intensity.

Prof. Weil is interested primarily in magnetic properties of materials below 1 K and in specific heat measurement of helium down to .02 K. Some work has been done in studying the effects of neutron radiation at cyrogenic temperatures on BaO and graphite as well as on liquid methane.

Centre d'Etudes Nucléares de Grenoble
Grenoble, Franch - June 17, 1963

Dr. Paul Perroud

A paper on heat transfer to liquid hydrogen in a high flux reactor was made available. Radiation studies on metals and graphite at liquid nitrogen temperature are in progress.

Le Centre D'Etudes des Matières Plastiques
Paris, Franch - June 19, 1963

Dr. P. Dubois

This laboratory is concerned with the wide scale evaluation of the physical (but not electrical) properties of plastics over a wide range of temperatures. The visit was conducted in the French language so that the impressions gained may not be complete. It appeared that only stress-strain measurements had been made at very low temperatures, i.e., liquid nitrogen. A non-published report was promised the author.

Institut Voor Lage Temperaturen en Technische Fysica
Leuven, Belgium - June 20, 1963

Prof. A. Van Itterbeek
Prof. H. Myncke
Dr. W. Van Dael

Prof. Van Itterbeek's laboratory has specialized in the study of liquid hydrogen. He was particularly interested in the G-E work on dielectric properties of cryogenic liquids. He described very old measurements of "static" dielectric constant determination and provided a copy of the original paper. Van Itterbeek knew of no other work on dissipation factor and of no other work on dielectric breakdown in liquid hydrogen. The ability of a magnetic field to produce dissymmetry in the liquid hydrogen molecule was mentioned. He believed that Keesom had obtained unpublished data on resistivity of liquid helium. The laboratory is presently not engaged in electrical studies except in respect to determining carrier mobility in Germanium at liquid helium temperatures.

A surprising amount of apparently outstanding highly theoretical work is being carried out by a relatively small staff in old buildings but with excellent equipment. In addition to physical studies of hydrogen and deuterium (i.e., absorption on glass, etc.), a great deal of work is underway at very low temperatures and with very high, pulsed magnetic fields (450,000 gauss for 1 to 10 millisec). Temperatures of .0001 ^{o}K can be held for 7 to 8 hours and 10^{-5} ^{o}K for about 20 seconds. Considerable attention is being directed to basic studies, such as nuclear spin and lattice interactions.

Van Itterbeek described techniques for purification of liquid hydrogen in which very pure gaseous hydrogen is introduced under 100 to 200 atmospheres pressure and condensed in a bundle of 30 μ dia. steel tubes (from Accles and Pollock in England). Small solid particles in the liquid hydrogen are trapped by the tubes. Helium, of course, is not separated in this fashion.

<u>N.V. Phillips Research Laboratories</u>
Eindhoven, Netherlands - June 21, 1963

Dr. J. Volger
Dr. Kohler
Dr. J.A. Kok

Dr. Volger is concerned with magnetic resonance, high field superconductivity and dielectric absorption at cryogenic temperatures. Because of the last interest, he was very much interested in the G-E work. His work started with the discovery of dielectric absorption peaks in glass at about 20 to 50^{o}K. His interest shifted to a study of quartz for which theory could more easily be developed. His theory, in brief, involves the ionic mobility of first element metals as related to "color centers" in the material. The theory is related to the idea of "Polorons" as first proposed by Fröhlich and developed by Holstein in the USA and Sewall in England. He believes Freyman in France has made the only other studies of dielectric absorption at cryogenic temperatures. He suscribes to the idea that unusual and unexpected dielectric properties may be uncovered at very low temperatures.

Dr. Kohler is primarily concerned with the development of heat engines and their counterparts (in reverse) for producing refrigeration. He has contractual relations with General Motors on heat engines and North American Phillips handles sale of air liquification units in the USA. A new, double stage, machine will produce 4 liters of liquid helium per hour, with a Carnot efficiency of 17%.

Dr. Kok is primarily concerned with dielectric breakdown in liquids and has done limited work with cryogenic liquids. He has summarized most of the known work on breakdown in cryogenic liquids as an appendix to a very new book (in German). He mentioned, also, the work of Young at MIT with liquid CO_2 near the critical point and the great variability found in its breakdown voltage. Kok believes that impurities and the influence of electrodes are very important in voltage breakdown studies of liquids. He has used a mixture of charcoal and Rochelle salt crystals to collect impurities in liquid helium.

<u>Laboratorium voor Technische Natuurkunde</u>
Delft, Netherlands - June 22, 1963

Prof. B.S. Blaisse
Prof. Jacobo M. Goldschvartz

Prof. Blaisse is making a major study of the electric strength of liquid helium both above and below the λ point and finds differences not reported by Edwards in the USA. He has discovered that electrode spacing may change or reverse the effect of temperature change on breakdown voltage. Breakdowns above and below the λ point cause different discharge patterns on the electrodes. Like Dr. Kok and others, Blaisse believes impurities and electrode characteristics are very important in cryogenic liquid breakdown tests. Liquid helium is purified by evaporation and subsequent condensation on activated charcoal followed by filtration below the λ point throught an unsintered Vycor, bacteriological filter. The filtration separates Helium III and absorbed water which is otherwise difficult to remove.

In addition to the breakdown studies, Blaisse is studying other cryogenic problems, such as thermal expansion with interferometer techniques (unpublished), double refraction in glass, and superconductivity phenomena.

Discussions with USSR Representatives to the IEC
Venice, Italy - May 27 to June 7, 1963

Informal discussions with representatives of the Soviet Union to IEC TC-15 on insulating materials indicated an interest and some activity on properties of insulating materials down to liquid nitrogen temperature with one reference to liquid helium and the problems of liquid oxygen. The extent of activity in the USSR was not determined and, likewise, details of the GE-NASA program were not discussed.